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American Society for Testing Materials

ORGANIZED IN 1898
INCORPORATED IN 1902

PROCEEDINGS, PART II

The Society is not responsible, as a body, for the statements and opinions advanced in this publication.

SUMMARY OF PROCEEDINGS OF THE PHILADELPHIA REGIONAL MEETING—SYMPOSIUM ON PAINT AND PAINT MATERIALS

The sixth regional meeting of the Society was held in Philadelphia, Pa., at The Warwick hotel on Wednesday, March 6, 1935, in conjunction with the Spring Group Meeting of A.S.T.M. Committees. Arrangements for the regional meeting were under the auspices of the Philadelphia District Committee.

The morning and afternoon sessions of the meeting comprised a technical program, a Symposium on Paint and Paint Materials sponsored by the Society's Committee D-1 on Preservative Coatings for Structural Materials, which assigned the development of the program to a special committee consisting of the following:

R. L. Hallett (*Chairman*), Chemist, National Lead Co.
Wayne R. Fuller, Technical Director, Pratt & Lambert, Inc.
H. A. Gardner, Chemical Engineer, The Institute of Paint and Varnish Research.
H. A. Nelson, Chief, Paint and Ceramic Research Division, New Jersey Zinc Co.
W. T. Pearce, In Charge of Sales Promotion and Development, The Resinous Products and Chemical Co., Inc.

The papers of the symposium cover various phases of the paint, varnish and lacquer industry and the several subjects are treated in a comprehensive, technical manner so that the papers and discussions should be of great interest to everyone, including consumers of paint, varnish and lacquer products, as well as those who produce them. The symposium consisted of the following 15 papers:

"Looking into the Future," by H. A. Gardner, Chemical Engineer, The Institute of Paint and Varnish Research

"Preparation, Use and Abuse of Specifications for Paint Materials," by P. H. Walker, Acting Chief, Chemistry Division, National Bureau of Standards

"Protective and Decorative Coatings for Railway Passenger Car Equipment," by A. M. Johnsen, Engineer of Tests and Chemist, The Pullman Co.

"Paint Testing," by C. D. Holley, Director of Paint Research, The Sherwin-Williams Co.

"Varnish Testing," by Wayne R. Fuller, Technical Director, Pratt & Lambert, Inc.

"Lacquer Testing," by H. E. Eastlack, Director, Parlin Laboratory, E. I. du Pont de Nemours and Co.

"Drying Oils," by S. O. Sorensen, Technical Director, Archer-Daniels-Midland Co.

"Zinc Pigments," by E. H. Bunce, General Manager, Technical Dept., The New Jersey Zinc Co.

"Lead Pigments," by R. L. Hallett and C. H. Rose, Chemists, National Lead Co., Brooklyn, N. Y.

"Titanium Pigments," by I. D. Hagar, Eastern Sales Manager, Titanium Pigment Co., Inc.

"The Mineral Earth Colors and Synthetic Iron Oxides," by J. W. Ayers, Director of Research, C. K. Williams and Co.

"Chemical Colors," by A. F. Brown, General Manager, Imperial Color Works

"Natural and Synthetic Resins," by W. T. Pearce, In Charge of Sales Promotion and Development, The Resinous Products and Chemical Co.

"Lacquer Solvents and Volatile Thinners," by R. M. Carter, Research Chemist, U. S. Industrial Alcohol Co.

"Turpentine and Petroleum Distillates as Thinners for Varnish and Paint," by J. M. Schantz, Manager, Technical Service, Naval Stores Dept., Hercules Powder Co.

The presiding officers of the sessions at the symposium were: Honorary chairman, G. B. Heckel, Editor-Publisher, *Drugs, Oils and Paints;* chairman, P. H. Walker, Acting Chief, Chemistry Division, National Bureau of Standards; and co-chairman, R. L. Hallett, Chemist, National Lead Co. The papers elicited a considerable amount of discussion which contributed greatly to the information contained in the papers. The papers, complete with discussion, have been published by the Society as a separate volume entitled, "Symposium on Paint and Paint Materials."

There were approximately 300 members and guests in attendance at the sessions.

The evening session, the Regional Meeting Dinner, which was of an informal nature, was addressed by Charles E. Bonine, Associate Director in Charge of Engineering, The Franklin Institute, in anticipation of a visit to The Franklin Institute Museum and Planetarium which followed the dinner.

AIRCRAFT: MATERIALS AND TESTING

EDGAR MARBURG LECTURE[1]

BY L. B. TUCKERMAN[2]

SYNOPSIS

This paper points out that all fields of science and technology have contributed to the development of the modern airplane. The discussion in the paper, however, is limited to the very narrow field of the relations between the mechanical properties of the material and the design of struts, beams, girders, stressed skin, etc., and the craft as a whole, to produce light-weight aircraft.

The major structural problem in aircraft is light-weight construction. The significance of light-weight construction is discussed by a comparison of power-driven vehicles, locomotive, steamship, automobile, airplane and airship on the basis of their structural density, their structural fineness ratio and the loads to which they are subjected.

It is pointed out that aircraft demand structures not merely lighter but of an entirely different order of lightness than had been necessary before. Success in meeting this demand has come from a combination of empirical design with theoretical investigations and materials and structural tests of a volume unprecedented in the history of any comparable structural field.

Theoretical and experimental investigations all show that the securing of light-weight construction depends upon spreading the material as far as possible out from the axis of the structure or its structural elements.

Typical light-weight construction is therefore thin-walled construction, that is, thin webs, thin flanges, thin-walled tubes, thin sheet surfaces, etc. The limit of the spreading is set by local instabilities: wrinkling, twisting, crumpling or other types of collapse, of the thin material. The problem is to secure the best possible balance between plastic yielding depending upon the strength-density ratio of the material, and major or local instabilities dependent upon its modulus-density ratio and finally the transitions between them dependent upon an accurate knowledge of its stress-strain curve.

These conclusions are illustrated by examples of simple structural members used in aircraft.

In the light of these conclusions the significant properties, density, strength-density ratio, and modulus-density ratio of selected materials are compared. The comparison shows that from these purely structural considerations, with the materials now available, there is little choice between the three types of construction now in use, wood, light aluminum alloys and high-strength alloy steels.

The choice between them must be based on other considerations, ease of fabrication, resistance to deterioration by weathering, etc., and finally upon knowledge of the designs which will utilize best the possibilities of the material.

Improvements in the quality of structural metals that increase the ease of fabri-

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[1] Read on June 26, 1935, before the Annual Meeting of the American Society for Testing Materials, Detroit, Mich.

[2] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

cation, and improvements in the technique of fabrication and joining of metal together by such processes as riveting, crimping, soldering, brazing and welding will make possible improved designs which are now impossible to fabricate.

With the best designs so far produced it is possible to make full use of the strength of materials already available only in relatively small parts of the structure. The modulus-density ratio is still the limiting property of the material and this ratio is practically unaltered by any treatment which can be given a material. Consequently no radical improvement in light-weight construction is at present to be expected from still further increasing the strength of the materials.

There is, however, much room for improvement in the knowledge of the possibility of designing to utilize more fully the strength of materials now available. Systematic series of tests on typical structural elements, covering ranges of materials, over-all dimensions, wall thickness and types of loading, sufficiently wide to determine all the types of instability and plastic yielding likely to occur in practical designs, to determine the limiting conditions of each type, and their relations to the stress-strain properties of the material as determined by parallel coupon tests—systematic tests such as these offer present promise of materially improved light-weight construction. Tests of this kind are being carried out in many laboratories, but many more are needed if progress in light-weight construction is to be maintained.

The principles, materials, and technique of light-weight construction developed chiefly in connection with aircraft are being increasingly used in other fields of construction, notably in light-weight railroad trains.

INTRODUCTION

The great longings of human beings are embodied in the most ancient myths which tradition has preserved in the songs and stories of all races. The fulfillment of the unsatisfied longings of men was the jealously guarded privilege of the gods and of men who became as gods. Woe betide mere man who impiously dared to aspire to share it. Zeus with his thunderbolt, Apollo with his chariot of fire, and Prometheus bound to the rock, tell us of the ages when men still longed in vain for the mastery of fire.

For how many æons has man longed to escape from

> "This inverted bowl we call the sky
> Whereunder, crawling, cooped; we live and die"?

How long has he cast envious eyes on the birds and thought with Dædalus:

> "Though the ways by land and sea be blocked,
> the paths of the air are open"?

This longing must be at least nearly as old as the longing for the mastery of fire and must be counted among men's great longings.

From time immemorial the gods had wings, and kings, like Tutankhamen, who became as gods wore wings as one of their most treasured attributes.

Over five thousand years ago the rashness of Etanah who on the wings of an eagle perished in daring the wrath of Ishtar was graven in everlasting stone. From a Grecian vase over two thousand years old, we learn of Icarus and his fate comes fresh to our eyes on the frescoed walls in the ruins of Pompeii. Yet the gods do relent. For untold centuries fire has been mere man's cherished possession and Vesta presides over the hearth.

In our day Darius Green is not struck dead for his impious words

"Birds can fly
An' why can't I?
Must we give in,"
Says he with a grin,
" 't the bluebird an' phoebe
Are smarter 'n we be?

"That Icarus
Was a silly cuss
His an' his daddy Dædalus,
They might 'a' knowed wings made o' wax
Wouldn't stan' sun-heat an' hard whacks,
I'll make mine o' luther
Er suthin' er other."

Instead he escapes with minor bruises.

How much serious study was given in the long ago to the problem of flight, we shall never know, but here and there an inkling appears. Most striking of all are the page on page of careful designs, Fig. 1, left to us in the sketch books of Leonardo da Vinci (1452-circ. 1488–1519).

It was, however, not to the birds that man owed his first real escape from the earth but to the dawn of the modern science of chemistry.

For over a century the hot air and gas balloons (1783) of Montgolfief and Charles aroused the enthusiasm of multitudes before the gliders or Lilienthal (1891) and Chanute (1896), the aerodrome of Langley (1903) and the aeroplane of the Wright brothers (1903) ushered in the modern era of flight.

We are living in an age which has seen the fulfillment of the dreams which men have dreamed for thousands of years. We have lived through its wonder and its glory, and even now it has become commonplace.

SCOPE OF DISCUSSION

Modern aircraft have been brought to their present state of development as the result of the cooperation of men in all fields of science and technology, mathematicians providing refined tools of analysis, physicists investigating the laws of air flow and the problems of elastic and plastic deformation of materials, chemists providing innumerable previously unknown materials, from dope on the airplane wing to the anti-knock in the engine fuel, metallurgists producing a wealth of new structural materials, aerodynamic engineers working out the detailed problems of efficient aerodynamic design, engine designers and builders making internal-combustion engines of previously undreamed lightness and reliability, structural engineers testing out the structural possibilities of materials, and finally designers, constructors and operators of aircraft synthesizing the work of all and putting the results to the test of practical use.

In limiting this paper to a very general discussion of the relations between the major structural problems of aircraft and the properties of the structural materials of which they are built, I do not wish to leave the impression that I consider these problems of greater or even as great importance as many others. They are the problems connected with aircraft with which I am most familiar, and they therefore loom larger in my thoughts than their relative importance warrants.

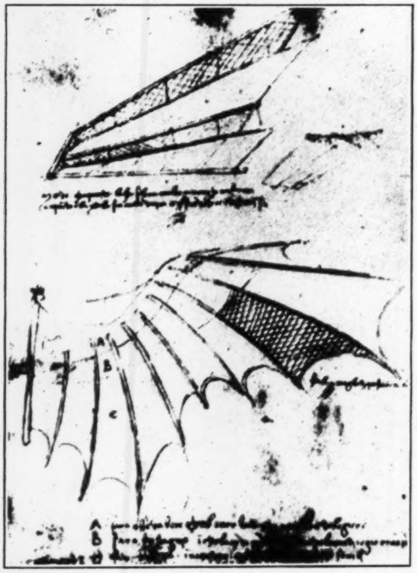

FIG. 1.—Design from the Sketch Books of Leonardo da Vinci.

Although the metallurgical problems involved in producing satisfactory structural metals for use in aircraft are of great interest, I shall mention them only incidentally. In this connection, I would refer to the excellent Symposium on Aircraft Materials held at the Thirty-third Annual Meeting and available in printed form.[1]

MEASURES OF LIGHT-WEIGHT CONSTRUCTION

You are all familiar with the statement that the basic structural problem in aircraft is light-weight construction. That is of course commonplace. Heavy things are not easily held up in the air. However "light-weight construction" is a very indefinite expression. Something, somewhat more definite is needed to serve as a basis for discussion. The dust that floats eastward from the drought-stricken regions in the middle west to hide the sun on the Atlantic seaboard and fall in a rain of mud is "light" enough to float in the air for days. Nevertheless when stirred into water it sinks to the bottom. Its "lightness" is mere smallness. Such lightness bears no relation to light-weight construction.

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[1] Symposium on Aircraft Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 26 (1930).

Structural Density:

A six million cubic foot airship weighing 200,000 lb. is from a structural standpoint as much a light-weight structure as a six hundred thousand cubic foot airship weighing 20,000 lb. The lightness of the construction lies in its "structural density" the ratio between the mass of the structure and the volume through which it is spread.

If all structures were as alike in general shape as are all airships and all were subjected to the same type of loads, a fairly close comparison of the lightness of their construction could be made on this basis. With structures of such widely different shapes as a locomotive, an ocean liner, an automobile, an airplane, and an airship, and subjected to such different forces in normal use, the comparison can only be rough. It is difficult to specify with any definiteness the volume which is structurally significant in all of these and it is impossible without going into detailed weight schedules to separate from the gross weight the structurally significant part as a net structural weight.

Structural Fineness Ratio:

There is still another factor which makes a comparison on the basis of volume and weight alone inadequate. Other things being equal a slender beam requires more structural material than a wide beam to carry the same load. Aerodynamic considerations force the designer to make the wings of an airplane relatively long and thin, a given structural density in an airplane wing represents therefore a greater achievement in light-weight construction than would the same structural density in an automobile body. Borrowing a word from airship nomenclature, the structural fineness ratio, the ratio of the length of the structure to its minimum structurally available breadth may be used as a measure of this modifying factor.

Character of Load:

Most difficult of all to evaluate are the differences in the forces to which structures are subjected in normal use, and the different allowance necessary to make for the possibilities of abnormal occurrences, but these must be taken into account if a fair comparison of lightness of construction is to be reached.

All of these considerations make it clear that there can be no generally valid measure of the lightness of a light-weight construction. Nevertheless, the comparison of power-driven vehicles on the basis of structural-density and structural-fineness ratio although at best it furnishes only the roughest sort of approximation shows differences great enough to give some definiteness to the conception of what is implied by light-weight construction in aircraft, Table I.

COMPARATIVE LIGHTNESS OF POWER-DRIVEN VEHICLES

In a standard locomotive about 500,000 lb. of material are built into a space of about 8600 cu. ft. giving an average density of 58.4 lb. per cu. ft., a specific gravity only slightly less than that of water. Before the days of automobiles and aircraft the "lightest weight" power-driven vehicle was the steamship. In a typical 500-ft. ocean liner the total volume is about four times the ship's displacement when empty so that the average specific gravity of such a ship is about one-fourth that of water. The "strength" of a ship however is not at all comparable to the strength of a locomotive.

TABLE I.—POWER-DRIVEN VEHICLES. COMPARATIVE STRUCTURAL-DENSITY AND STRUCTURAL-FINENESS RATIO.

Structure	Weight, lb.	Volume, cu. ft.	Structural Density, lb. per cu. ft.	Structural Specific Gravity	Structural-Fineness Ratio
(water)	....	....	62.4	1.00	....
Locomotive	502 000	8 600	58.4	0.94	5.9
Ocean liner	20 000 000	1 330 000	15.0	0.24	12.5
Passenger automobile	2 700–3 000	150–300	10.0–20.0	0.16–0.32	2.4–3.5
Transport monoplane:					
Total structure	25 000	5 000	5.0	0.080	6.5
Wings	2 900	2 100	1.38	0.022	28.6
Pursuit monoplane:					
Total structure	2 300	220	10.5	0.168	3.9
Wings	300	50	6.0	0.096	46.4
("standard" air)	....	....	0.07635	0.00122	....
Airship	221 000	7 400 000	0.030	0.00048	5.9
Stratosphere balloon	6 350	3 700 000	0.0017	0.000027	1.0

If we could conceive of supporting such a ship on four wheels for travel on land and place the wheels approximately in the same relative position as the wheels of an automobile, calculations would show that the ship would break in two under its load.

Fortunately the naval designer does not need to provide for such conditions of support. The sea furnishes an ideally continuous support, so that the worst he has to contend with are his standard waves, severe as they may be in times of storm, with their shifting support and their buffetings.

If it had been necessary for the naval designer to contend with shocks and loads and concentrated supports at all comparable in relative magnitude to those which daily fall to the lot of railroad locomotives and automobiles, his problem of light-weight construction would have been even more difficult than it has been, and the era of typical light-weight construction would have been inaugurated much earlier.

In a modern passenger automobile some 3000 lb. of material are built into a space of 150 to 300 cu. ft. giving an average density of from 10 to 20 lb. per cu. ft., a specific gravity from one-sixth to one-third that of water. These figures alone would indicate that the automobile and the ocean liner had approximately the same "lightness of construction." Actually, because of the relatively greater forces to which it is subjected the structure of the automobile must be classed as of relatively lighter weight construction. How much allowance should be made it is difficult to estimate.

In the modern airplane we find a type of construction of still another order of lightness. The inertial loads to which it is subjected when pulling out of a steep dive or under the impacts of landing or taxying at speeds as great as 70 m.p.h. are certainly relatively as severe if not more severe than any which fall to the lot of an automobile in normal use so that in that respect they may perhaps be considered roughly comparable. There is built in a transport monoplane, with a volume of about 5000 cu. ft., only 25,000 lb. of material, an average density of only about 5 lb. per cu. ft., about one-half that in automobiles of the lightest construction. With this low allowance of weight it must endure stresses relatively at least as great and probably much greater.

As was noted before, using the same materials, the strength which it is possible to build into a structure to resist bending moments depends in large measure upon its structural-fineness ratio; the ratio of its longest to its shortest structurally significant dimension.

In naval construction, fineness ratios of the order of 12 are usual, and ships are subjected to relatively smaller forces. In automobiles, where the forces are perhaps more nearly comparable, fineness ratios of the order of 3 or 4 are usual. The automobile designer does not make full use of the possibilities implied by these ratios but they are available if he should wish to do so.

Contrast these with the wing structure of a modern transport monoplane with a weight of 2900 lb., a volume of 2100 cu. ft., a density of 1.38 lb. per cu. ft., less than one-seventh that of the lightest automobile, with a wing span of 90 ft. and a wing thickness at the root of only 3 ft., 2 in., a fineness ratio of 28.6. The fact that practically all of the load must be carried by these wings and practically none by the whole area of the fuselage still further increases the relative magnitude of the stresses. Further the airplane user constantly asks for still lighter construction.

The difficulty of obtaining a fair comparison of the lightness of a structure on this basis may be emphasized by contrasting a pursuit monoplane with a transport monoplane (Table I). About one-tenth as much mass is concentrated in about one-twentieth the volume making the average density about twice as great. The details of the design data, however, show that in the pursuit plane the power plant makes up about 44 per cent (1000 lb.) of the weight, as contrasted with about 16 per cent (4000 lb.) in the transport so that the strictly structural densities (5.7 lb. per cu. ft. and 4.2 lb. per cu. ft.) do not differ so widely. The wing densities are in striking contrast, the wing of the pursuit plane being over four times as dense. However, its fineness ratio is over 60 per cent greater and in addition it is expected to survive acrobatic maneuvers in combat flying, which would surely wreck the transport plane. Taking these differences into consideration it might well be that this pursuit monoplane was effectively of "lighter weight construction" than the transport. The structural density alone gives a very incomplete comparison.

Finally let us look for a moment at the lightest of light-weight construction in power-driven vehicles—the modern airship. A total dead weight of 221,000 lb. spread out in a volume of 7,400,000 cu. ft., an average density of 0.030 lb. per cu. ft., a specific gravity less than 0.0005 that of water and less than half the density of air. It is true that the fineness ratio of such a ship is only about 6, although some fairly successful ships have been built with ratios as high as 12.

It is true also that an airship normally lands at very low speeds and consequently does not need to be designed to stand up under impact loads comparable to those which an airplane must sustain. In these respects the airship designer is fortunate, for even with these relatively favorable conditions he has no easy problem in designing a safe structure under such severe weight limitations.

The only large land structure at all comparable to an airship in lightness with which I am familiar is the "big tent" of the circus. Even that is not comparable since it relies upon the millions of tons of mother earth as one of its main structural elements, and the circus manager would not willingly see it fly through the air.

Because of my personal interest in the comparison I have added as lagniappe the data on the stratosphere balloon Explorer II. Like the flowers that bloom in the spring tra la, they have nothing to do with the case.

In spite of the admitted inadequacies of the comparisons which these figures furnish, the contrasts still are sufficiently great to be of real significance.

LIGHT-WEIGHT CONSTRUCTION THE BASIC PROBLEM IN AIRCRAFT

The basic structural problem which confronted the pioneer aircraft industry—light-weight construction—was, therefore, not merely the problem of building structures somewhat lighter than comparable structures in current use. It was the problem of building safe and strong structures of an entirely different order of lightness.

The adequate solution of this problem required the recognition and development of the structural possibilities of materials and the utilization of these possibilities far beyond any precedents.

Empirical extrapolation from existing structures was the method by which, for centuries, men built new and improved structures. Even after the foundation of a basic theory of structures by the mathematicians, mathematical physicists and far-sighted engineers of the eighteenth and first half of the nineteenth century, the greater part of structural development was still by mere empirical extrapolation. Theoretical studies and tests were few and far between.

Empirical extrapolation is a slow process. New structures are built departing in some measure from existing structures. If they fail a new attempt is made until by trial and error success is achieved. Wide departures from well-tried structures, either in size or design, are all too likely to fail. Not infrequently in the history of engineering, disaster has followed extrapolations to new structures somewhat outside the range of previous experience, yet differing far less from existing successful structures than would meet the demands of aircraft. To many the Quebec Bridge is still a vivid memory.

Much brilliant empiricism and the lessons of many failures have played their part in the development of the light-weight structure of aircraft, but the rapid developments of the past 30 yr. could not and have not come from these alone. Theoretical investigations, and materials and structural testing, of a volume unprecedented in the history of engineering, have given us far more knowledge of the properties of materials, and the relationships between these properties and the geometrical shape of a structure which determine its strength. By far the greater part of this advance has come from the needs of light-weight construction in aircraft.

THIN-WALLED CONSTRUCTION IS THE SOLUTION

If structures were subjected to tensile forces only, the problem of light-weight construction would be simple. Unfortunately that is not possible. In any self-contained structure, even if it were required to sustain no external forces, tensile forces must be balanced against compressive forces, and all these structures must sustain a variety of compressive and bending loads. The major difficulties arise from these.

Long before men came upon the earth Nature met and solved the problem by two basic inventions. The exoskeleton of the flying insects, Fig. 2, is the prototype of monocoque or stressed skin construction, and the hollow bones of the endoskeleton of the flying reptiles, Fig. 3, birds and

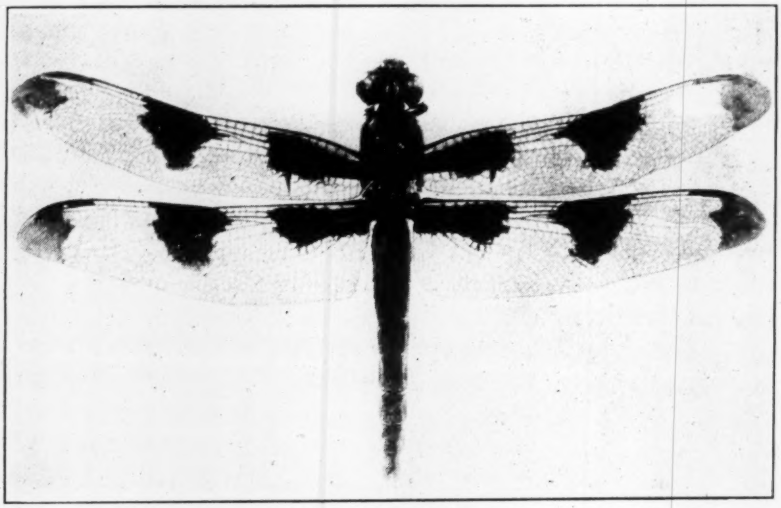

FIG. 2.—Dragon Fly.

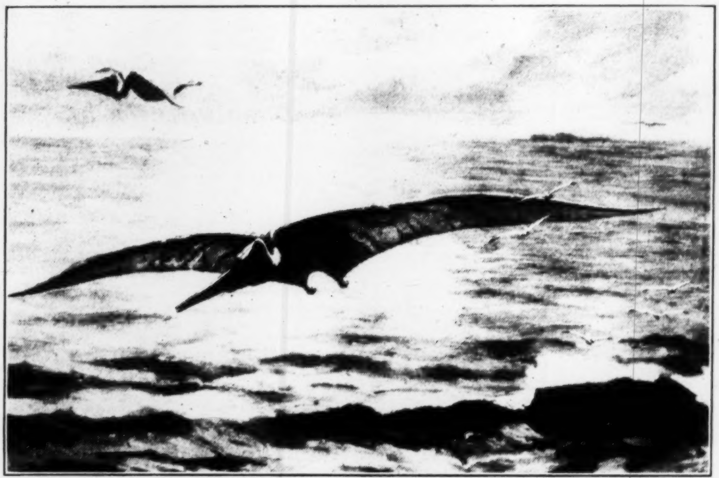

FIG. 3.—Reconstruction of Pteranodon.

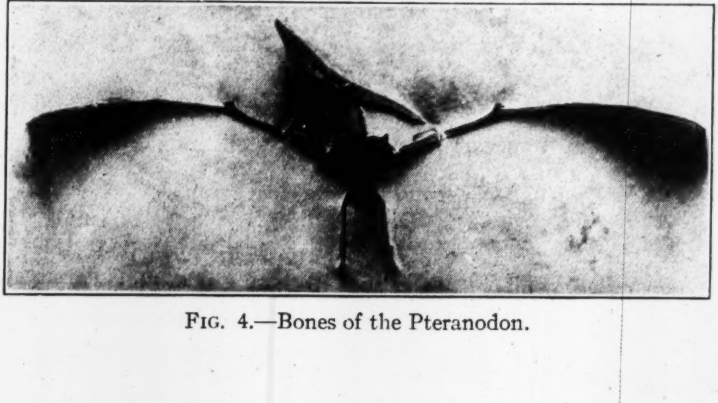

FIG. 4.—Bones of the Pteranodon.

mammals are the prototypes of tubular constructions (Fig. 4). Both of these are illustrations of the geometric requirement of light-weight construction to sustain bending and compressive forces. This consists in spreading the material as far out as possible from the axis of the structure or its structural elements and at the same time holding it firmly together. The result is thin-walled construction. If this spreading of the material and thinning of the walls can be carried far enough, the limiting strength of the structure is set by the strength of the material. This was the case in the greater portion of the heavy construction of the days preceding aircraft.

CRITICAL INSTABILITY LIMITS THE WALL THINNESS

Even in heavy construction, particularly in the case of columns, it was not always found possible to spread the material sufficiently far from the structural axis to reach this limit. An earlier limit was set by the phenomenon of critical instability.

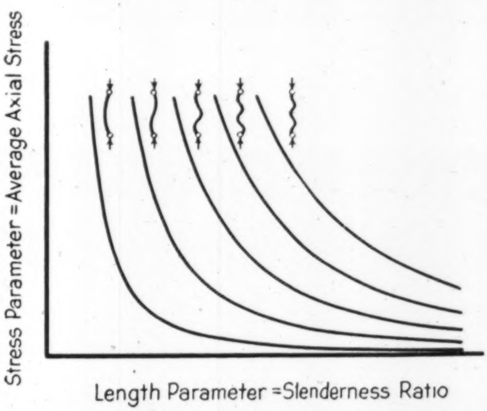

FIG. 5.—Critical Elastic Instability of Long, Slender Euler Columns.

This phenomenon, of secondary importance in heavy construction, has proved to be the major structural problem in the light-weight construction of aircraft. The progress made in the last few decades in the knowledge of structures has been largely a fuller understanding of the conditions which determine critical instability.

Probably the type of critical instability with which men first become familiar has been a commonplace experience for many thousands of years, yet is one which few, even today, think of as a phenomenon of this kind. I refer to the familiar kinking of twisted ropes, cords, strings and thread. Under definite relationships of torsional moment and axial forces which in themselves have no bending moment the structure becomes critically unstable and bends into a helix. This same type of instability is sometimes of importance in the torsion of shafts. It was not until 1744 when Euler[1] published his epoch-making paper, that anyone grasped the essential nature of such phenomena.

We are all familiar with the theory of the slender Euler column, Fig. 5. If the first type of failure by bending as a unit is prevented by restraints at the middle, the load can be increased above the first "Euler" load until at the second "Euler" load (4 times as big) it buckles in a two-bay form. If restraints are placed at the third point the loads can be carried still higher to the third Euler load, and so on.

We gain a better conception of relationships involved from the curves in which the average axial stress at failure through instability is plotted as ordinate with the slenderness ratio as abscissae. This, the most common type of critical instability in "heavy" construction, is the simplest of all in its relationships and was for over a century the only type about which men had any real knowledge.

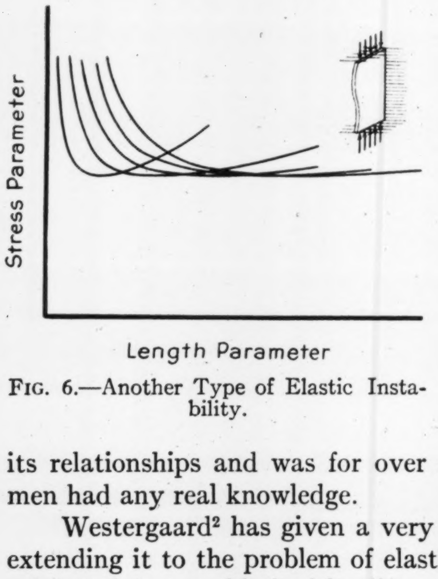

FIG. 6.—Another Type of Elastic Instability.

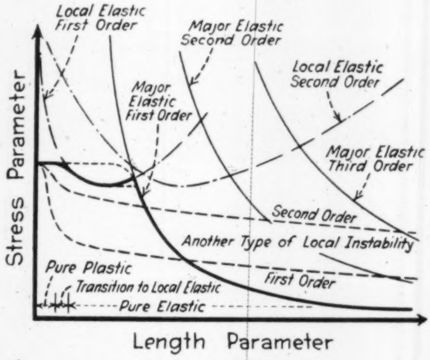

FIG. 7.—Schematic Diagram of General Case of Critical Instability.

Westergaard[2] has given a very complete generalization of this theory extending it to the problem of elastic instability of any shape of structure subjected to any kind of loading. For each structure and each type of loading there are, as in the Euler column, theoretically an infinite number of types of critical instability each with its own critical load. These types of instability can usually be grouped in families having some common characteristic such as the family of Euler instabilities. In general the critical loads of such a family (Fig. 6) are not so simply related as are the successive Euler loads on a column, but change in entirely different and unrelated ways with changes in the shape of the structure or type of loading. Even less regularity can be found between the critical loads of different families. When loaded in the prescribed manner the structure will become unstable at the lowest of these loads (Fig. 7) and fail, provided it has not failed at a lower load by plastic yielding or brittle breaking of the material.

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[1] Leonhard Euler, "Methodus Inveniendi Lineas Curvas Maximi Minimive Proprietate Gaudentes," pp. 245–270, Bousquet and Co., Lausanne and Geneva (1744).

[2] H. M. Westergaard, "Buckling of Elastic Structures," *Transactions*, Am. Soc. Civil Engrs., Vol. 85, pp. 576–676 (1922).

Westergaard's theory gives us a clear conception of the phenomenon with all its manifold possibilities, and serves as an excellent guide in the planning of structural tests. In some cases it is of use in detailed calculations of specific types of instability, but usually its very generality makes it difficult to apply and special cases are found to yield more readily to special treatment. For a considerable number of special cases fairly adequate theories have been worked out for small deformations within the elastic range. To enumerate them would be tedious but I might mention some of the men to whom we owe this knowledge—Engesser,[1] Considère,[2] Prandtl,[3] Reissner,[4] Bryan,[5] Southwell,[6] von Kármán[7] and Timoshenko[8]—and a host of others.

APPROXIMATE QUANTITATIVE FORMULATION OF THE PROBLEM

So far we have only considered the problem of critical instability from a qualitative standpoint. The quantitative problem of determining the relative suitability of different materials in light-weight construction makes it necessary to indulge in a certain amount of mathematics. It is necessary to determine in broad outline the relationships between the mechanical properties of the materials and the strength of structures which can be built from them. Since the problem we are discussing is the problem of light-weight construction, the controlling factor is the strength-weight ratio of the structure, the ratio between the load P or the torque T which it can safely carry and the mass M of the material which is necessary to construct it.

No general formulation of the problem is possible since the types of load to be carried and the shapes of the structures which carry them are infinite in their variability. However, it is possible to consider with some degree of definiteness the factors involved in typical structural elements, tension members, torsion members, beams and compression members, and from the information gained try to understand something of the general relationships.

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[1] Fr. Engesser, "Über die Bestimmung der Knickfestigkeit gegliederter Staebe," *Zeitschrift* des Oesterreichischen, Ingenieure und Architekten Verein, Vol. 65, pp. 769–772 (1913).

[2] A. Considère, "Resistance des Pièces Comprimées," Congrès International des Procédés de Construction, Paris, 1889, *Comptes Rendus*, Baudry, Paris, pp. 371–397 (1891).

[3] Ludwig Prandtl, "Kipperscheinungen. Ein Fall von instabilem elastischen Gleichgewicht," Stich, Nuernberg (1899).

[4] H. Reissner, "Über die Knicksicherheit ebener Bleche," *Zentralblatt der Bauverwaltung*, Vol. 29, pp. 93–96 (1909).

[5] G. H. Bryan, "On the Stability of a Plane Plate Under Thrusts in Its Own Plane," *Proceedings*, London Mathematical Soc., Vol. 22, pp. 54–67 (1890).

[6] R. V. Southwell, "On the General Theory of Elastic Stability," *Philosophical Transactions*, Royal Soc. (London), A, Vol. 213, pp. 187–244 (1913).

[7] Th. von Kármán, "Analysis of Some Typical Thin-Walled Structures," *Transactions*, Am. Soc. Mechanical Engrs., Aeronautical Engineering, Vol. 5, pp. 155–158 (1933).

[8] S. Timoshenko, "Sur la Stabilité des Systèmes Élastiques," *Annales des Ponts et Chaussées*, Series 9, Vol. XV, pp. 496–566; Vol. XVI, pp. 73–132; Vol. XVII, pp. 372–412 (1913).

FAILURE BY FAILURE OF MATERIAL

STRENGTH-DENSITY RATIO OF THE MATERIAL A LIMITING FACTOR

The relationships between the properties of materials and the possibilities of light-weight construction of members subject to pure tensile stresses is simple. We have the basic formula:

$$\frac{P}{A} = S$$

where P = the load the structure can carry,
A = the sectional area of the member, and
S = the stress which the material can safely stand.

Remembering that

$$M = d \cdot L \cdot A$$

where M = the mass,
d = the density of the material,
L = the length, and
A = the cross-sectional area,

the strength-weight ratio of a tension member is given by

$$\frac{P}{M} = \frac{1}{L}\frac{S}{d}$$

Ignoring the problem of terminal fittings, which, however, frequently arises to plague the designer, we see that the strength-weight ratio of the member is directly proportional to the strength-density ratio of the material, inversely proportional to the length of the member and is wholly independent of the shape of the section.

All of the material in the member is uniformly stressed to its limit so that this represents the maximum strength-weight ratio that can be reached in any structure built of material with this same strength-density ratio.

This is the only simple problem in the whole of structural designing. For all the rest we enter into a field of enormous complexity involving the geometrical properties of the most varied cross-sections, combined with the elastic and other properties of the material.

However, it is possible from this complexity to sort out some general relationships to serve as rough guides in design. These relationships involve, in addition to the type of loading, the properties of the material and the details of the design, geometrical factors involving the over-all dimensions of the sections, and factors involving the minimum thickness of the material in the sections. To present these relationships in all their generality would lead into a maze of mathematics which would bewilder rather than clarify. For that reason I have chosen to present the problem of the simplest geometrical shape which contains these two essential geometrical factors. This is the circular tube. The significant structural factors are shown in Fig. 8.

Considering such a tube used as a beam (Fig. 9) we see that the strength-weight ratio, P/M, of the beam can be expressed in the form:

$$\frac{P}{M} = \left(\frac{1}{L}\frac{S}{d}\right)k_2 \frac{\sqrt{A}}{L}\frac{1}{(t/R)^{\frac{1}{2}}}$$

The quantity within the parentheses is the same as the strength-weight ratio of a tension member. The formula contains in addition the three factors k_2, $\frac{\sqrt{A}}{L}$ and $\frac{1}{(t/R)^{\frac{1}{2}}}$. The first of these, k_2, is a dimensionless factor depending upon the end conditions and load distribution and cannot be altered without altering the nature of the whole structure or the type of its loading. The second $\frac{\sqrt{A}}{L}$ is in the nature of a "shape factor" but one which cannot be increased without increasing the cross-sectional area which would increase the weight of the beam, or by decreasing the length which would again alter the nature of the whole structure. The third factor, however, dependent upon the "wall thickness" ratio t/R, can be altered at will without altering either the mass, the length, end conditions or the load distribution. Taking the formula at its face value it indicates that by diminishing the wall thickness and correspondingly increasing the radius to keep the area constant the strength-weight ratio of the tube used as a beam can be increased indefinitely.

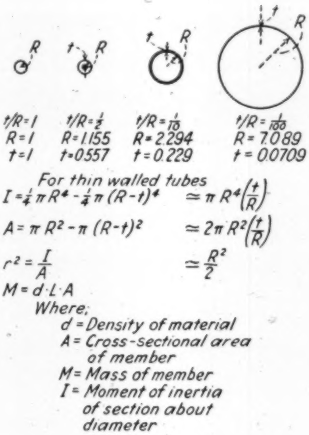

R
t
R
t
R
t
R
$t/R=1$
$R=1$
$t=1$
$t/R=\frac{1}{2}$
$R=1.155$
$t=0.557$
$t/R=\frac{1}{10}$
$R=2.294$
$t=0.229$
$t/R=\frac{1}{100}$
$R=7.089$
$t=0.0709$
For thin walled tubes
$I=\frac{1}{4}\pi R^4-\frac{1}{4}\pi(R-t)^4 \simeq \pi R^4\left(\frac{t}{R}\right)$
$A=\pi R^2-\pi(R-t)^2 \simeq 2\pi R^2\left(\frac{t}{R}\right)$
$r^2=\frac{I}{A} \simeq \frac{R^2}{2}$
$M=d\cdot L\cdot A$
Where;
d = Density of material
A = Cross-sectional area of member
M = Mass of member
I = Moment of inertia of section about diameter

FIG. 8.—Structure Factors of Circular Tubes of Given Area.

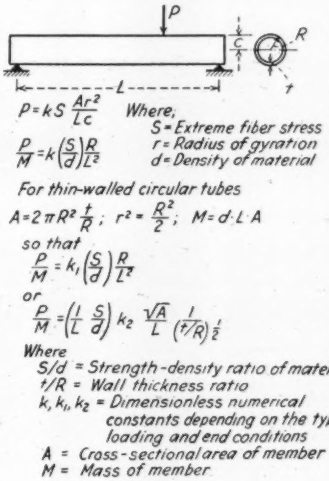
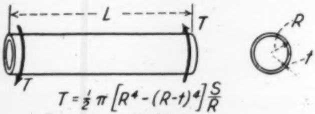

P
c
R
t
L
$P=kS\frac{Ar^2}{Lc}$
Where;
S = Extreme fiber stress
r = Radius of gyration
d = Density of material
$\frac{P}{M}=k\left(\frac{S}{d}\right)\frac{R}{L^2}$
For thin-walled circular tubes
$A=2\pi R^2\frac{t}{R}$; $r^2=\frac{R^2}{2}$; $M=d\cdot L\cdot A$
so that
$\frac{P}{M}=k_1\left(\frac{S}{d}\right)\frac{R}{L^2}$
or
$\frac{P}{M}=\left(\frac{1}{L}\frac{S}{d}\right)k_2\frac{\sqrt{A}}{L}\frac{1}{(t/R)^{\frac{1}{2}}}$
Where
S/d = Strength-density ratio of material
t/R = Wall thickness ratio
k, k_1, k_2 = Dimensionless numerical constants depending on the type of loading and end conditions
A = Cross-sectional area of member
M = Mass of member

FIG. 9.—Strength-Weight Ratio in Circular Tubular Beams.

With the aid of much mathematics this kind of reasoning can be extended to cover all kinds of beams of all sorts of shapes of section. The wall thickness ratio, t/R will be replaced by other "wall thickness" ratios such as ratios of web thickness to web breadth, flange thickness to flange width, cover plate thickness to rivet spacing, slenderness ratio of chord members between lattice points, etc., and in complicated sections not one but several "wall thickness" ratios will be involved. Further, instead of the simple function $(t/R)^{\frac{1}{2}}$, there will enter more complicated functions, but in all cases continuous increasing functions of the wall thickness ratios.

The general picture, however, remains unchanged; taken at its face value all this reasoning leads to the conclusion that for the same weight of material of the same strength-density ratio increase of over-all dimensions of the cross-section of a beam with decrease of wall thickness leads to a greater strength-weight ratio of the beam and consequently to more economical use of the material and to lighter weight construction.



$$T=\tfrac{1}{2}\pi\left[R^4-(R-t)^4\right]\frac{S}{R}$$

For thin-walled tube

$$A=2\pi R^2\frac{t}{R},\ M=d\cdot L\cdot A$$

and

$$T=2\pi SR^2t$$

or

$$\frac{T}{M}=\left(\frac{1}{L}\,\frac{S}{d}\right)k\sqrt{A}\,\frac{1}{(t/R)^{\frac{1}{2}}}$$

Where
T = Torque
S/d = Strength-density ratio
t/R = Wall thickness ratio
k = Dimensionless numerical constant
d = Density of material
A = Cross-sectional area of member
M = Mass of member

FIG. 10.—Strength-Weight Ratio of a Circular Tube in Torsion.

We know, of course, that there is a limit beyond which this increase of strength cannot be carried. If the lateral dimensions become comparable with the length, the simple Euler-Bernoulli theory upon which the formula is based ceases to be an adequate approximation, and in no case can the strength-weight ratio of the simple tension member be exceeded. This limit, however, is far outside the range of any practical construction and does not enter into the present discussion. Another limit usually lower and important for this discussion is set by the fact that if the walls become too thin they will crumple or otherwise become unstable in detail. Up to these limits, however, whatever the strength-density ratio of the material, for a given weight the beam is stronger the lighter its walls and the greater its radius.

The general problem of the torsional strength of structures is much more complicated than the problem of beams, but for the closed circular tube the problem is even simpler (Fig. 10).

Except for the fact that the weight increase is proportional to the length instead of to the square of the length, the equation is identical in form with that for the beam. The stress S which the material of the twisted tube can safely withstand has however a different significance; it is a shearing stress, instead of a tensile or compressive stress as in the case of the beam. For a general discussion, however, this difference does not need special consideration. The ratio between the shearing, tensile, and compressive stresses which the metals used as main structural elements can safely withstand is roughly the same, and the differences need to be taken into account only in an accurate study of specific structures.

No broad generalization, such as was stated for beams, covering all sorts of shapes of section is possible for structures in torsion. To make this clear, it is only necessary to contrast the great torsional strength of a closed circular tube, with the pronounced weakness in torsion of the same tube slit down the side. Nevertheless, with the aid of very much mathematics, it is possible to make a rougher limited generalization to cover the shapes of sections that have any practical value in resisting torsion. Broadly speaking these are closed sections with a periphery everywhere practically convex and with minimum and maximum lateral dimensions not too widely different.

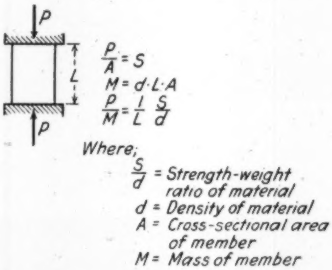

P
L
P
$\frac{P}{A} = S$
$M = d \cdot L \cdot A$
$\frac{P}{M} = \frac{1}{L} \frac{S}{d}$
Where;
$\frac{S}{d}$ = Strength-weight ratio of material
d = Density of material
A = Cross-sectional area of member
M = Mass of member

FIG. 11.—Strength-Weight Ratio in Short, Stocky Compression Members.

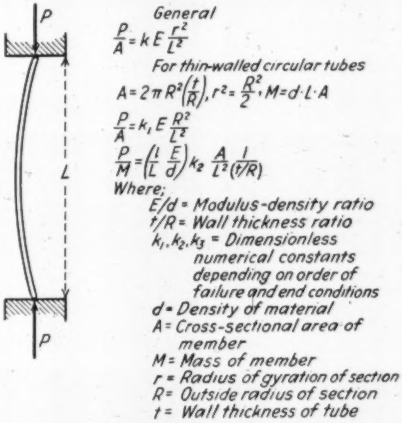

P
L
P
General
$\frac{P}{A} = kE\frac{r^2}{L^2}$
For thin-walled circular tubes
$A = 2\pi R^2\left(\frac{t}{R}\right), r^2 = \frac{R^2}{2}, M = d \cdot L \cdot A$
$\frac{P}{A} = k_1 E \frac{R^2}{L^2}$
$\frac{P}{M} = \left(\frac{1}{L}\frac{E}{d}\right) k_2 \frac{A}{L^2} \frac{1}{(t/R)}$
Where;
E/d = Modulus-density ratio
t/R = Wall thickness ratio
k_1, k_2, k_3 = Dimensionless numerical constants depending on order of failure and end conditions
d = Density of material
A = Cross-sectional area of member
M = Mass of member
r = Radius of gyration of section
R = Outside radius of section
t = Wall thickness of tube

FIG. 12.—Strength-Weight Ratio in Circular Tubular Euler Columns.

For such sections, with appropriate mental reservations to exclude possible freak shapes, the reasoning taken at its face value again leads to the conclusion that, for the same weight of material of the same strength-density ratio, increase of over-all dimensions of the cross-section of a torsion member with decrease of its wall thickness leads to a greater strength-weight ratio of the member and consequently to more economical use of the material and lighter weight construction.

So far only two material properties have entered into the formulas: *S*, the stress which the material can safely withstand and *d*, its density, combined in the strength-density ratio *S/d* of the material to which the strength-weight ratio of the structure is proportional.

FAILURE BY MAJOR CRITICAL INSTABILITY

MODULUS-DENSITY RATIO OF THE MATERIAL A LIMITING FACTOR

With purely axial compressive loads the picture changes. For very short and stocky members we merely have to repeat the formulas for tension members (Fig. 11) but for long slender Euler columns critical instability is the controlling factor (Fig. 12).

According to this formula the strength-weight ratio of the column does not depend at all upon the strength-density ratio of the material but instead is proportional to its modulus-density ratio and, as in the case of the beam, also upon the shape of the section as expressed by the wall thickness ratio (t/R), appearing again in the denominator but without the square root sign.

As in the case of the beam, with the aid of much mathematics this type of reasoning can be extended to cover all kinds of columns of all kinds of shapes. In details the picture can frequently be materially improved. If lateral loads are not present, a tapered column may be used increasing somewhat the value of k_2. Lattice or box-type columns may be used concentrating thicker material locally and using thinner material merely to resist the shearing stresses. Various degrees of fixity may be used at the ends or light-weight lateral supports may be used to prevent the lower types of Euler failure. However, whenever it is found possible to carry through calculations for any of these shapes in detail the result is a formula essentially identical in content with this, although frequently improved by a somewhat higher value of k_2, and involving as in the case of the beam other wall thickness ratios entering the formulas in the denominator as continuous increasing functions of these ratios.

Again the general picture remains unchanged. Taken at its face value the reasoning leads to the conclusion that regardless of the strength-density ratio of the material, for the same weight of material of the same modulus-density ratio, increase of the over-all dimensions of the cross-section of an Euler column, with decrease of wall thickness, leads to a greater strength-weight ratio of the column and consequently to more economical use of the material and lighter weight construction.

We know of course, as in the case of the beam, that the strength cannot be increased indefinitely by continually decreasing the wall thickness. In the column, however, not one but either one of two distinct types of failure may set the limit (Fig. 7). The wall thickness may be decreased so much that crumpling or other detailed failure enters. On the other hand before that happens the stresses in the material may have risen higher than the material can safely stand and the failure is determined by conditions intermediate between the Euler failure dependent solely upon the modulus-density ratio and the failure of a short stocky compression member dependent upon the strength-density ratio of the material.

There are other types of critical instability of beams and columns as a whole which are not found in the circular sections; such for example are the tipping instability of a narrow beam under edge load and the twisting instability of a column as a whole under axial compression. In broad outlines their theory parallels that of the ordinary column as outlined above. When the beam or column is long enough, its strength-weight ratio is independent of the strength-density ratio and depends instead upon a modulus-density ratio of the material and wall thickness ratios. These types of instability are only infrequently major factors in a design. To discuss them all in detail would take too long. The discussion of any one would merely repeat with a few changed words the discussion of the ordinary column failures, and would add nothing new in principle.

LOCAL INSTABILITY

To complete this general picture of the relationship between the properties of materials and the possibilities of light-weight construction, some idea must be gained of the conditions which determine the local crumpling or other local instabilities of small portions of thin-walled structures. These instabilities are of bewildering diversity. There are the symmetrical (Fig. 13) and lobate (Fig. 14) crumpling of tubes under axial load, the local buckling of outstanding edges of open sections (Fig. 15) the buckling (Fig. 16) or twisting (Fig. 17) of open-section chord members between lattices, the buckling of web members, different in character for compression (Fig. 18) and shear (Fig. 19), the elliptical buckling of a bent circular tube, the diagonal buckling of the wall of a tube under transverse shear and bending (Fig. 20), collapse of a tube wall under external pressure (Fig. 21), wrinkling of a thin sheet under shear (Fig. 22), and others too numerous to mention.

The distinction between instabilities of a structure or a structural member as a whole such as the Euler column instability which may be called "major instabilities" and the local instabilities of a small portion or a number of small portions of the structure or member is of course wholly a matter of convenience in thought. All of them with no exceptions are included under Westergaard's all embracing theory and from this general viewpoint are not distinct in character but merely different manifestations of the same phenomenon. Nevertheless the distinction is necessary to a clear understanding of the relations of elastic instability of structures.

There is no geometrical distinction by which one single dimension of

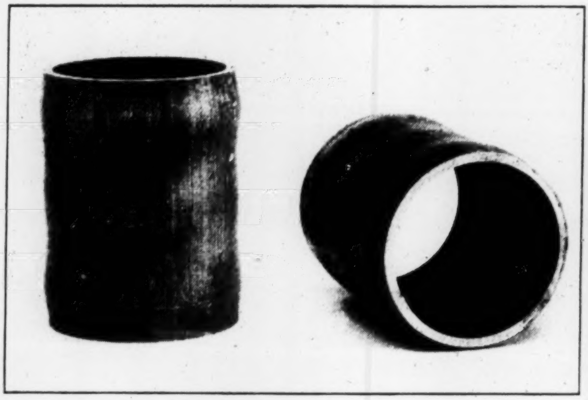


FIG. 13.—Symmetrical Instability.

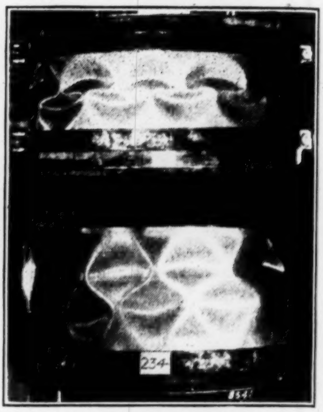


FIG. 14.—Lobate Instability.

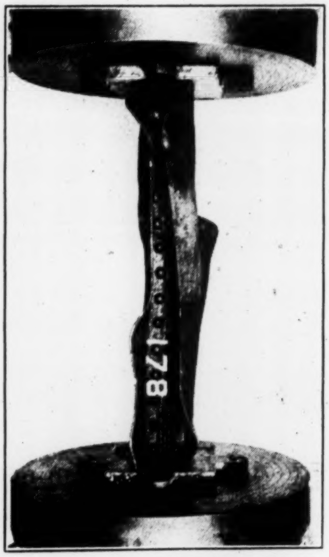


FIG. 15.—Local Buckling of Outstanding Edges of Open Sections.

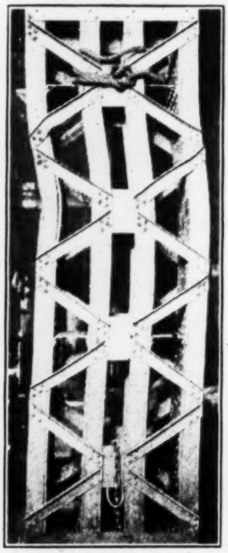


FIG. 16.—Buckling of Open Section Cord Members Between Lattices.

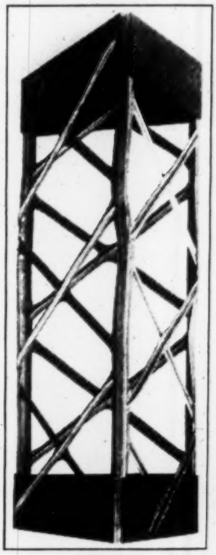


FIG. 17.—Twisting of Open Section Cord Members Between Lattices.

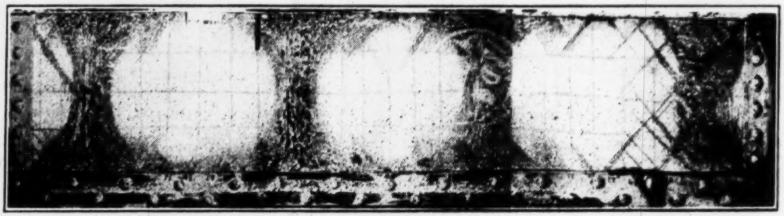


FIG. 18.—Buckling of Web Members in Compression.

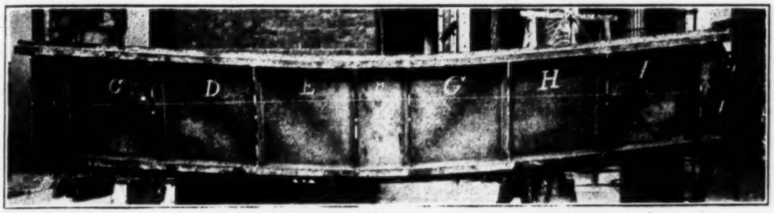


FIG. 19.—Buckling of Web Members in Shear.

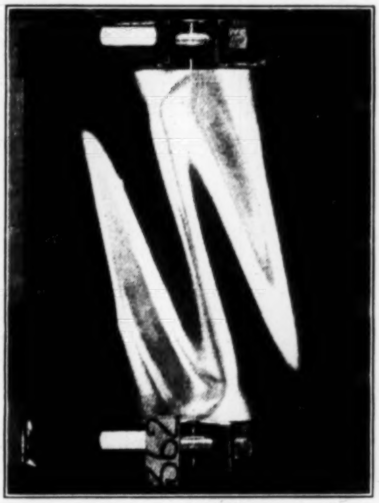


FIG. 20.—Diagonal Buckling of the Wall of a Tube Under Transverse Shear and Bending.

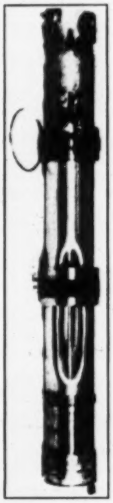


FIG. 21.—Collapse of a Tube Wall Under External Pressure.

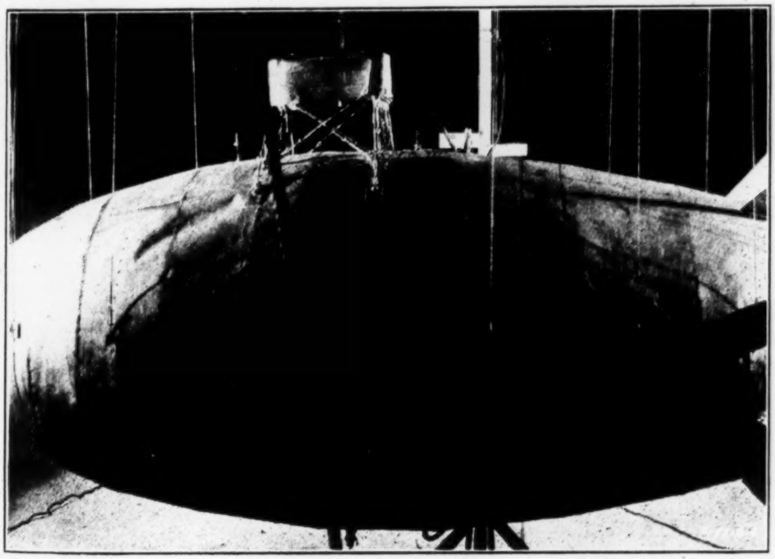


FIG. 22.—Wrinkling of Thin Sheet Under Shear.

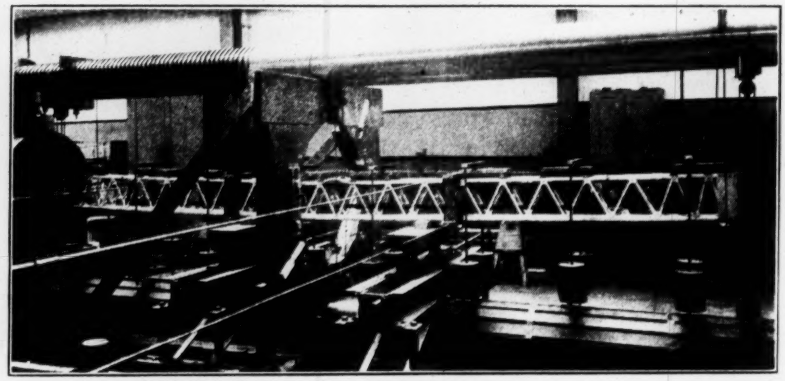


FIG. 23.—Twisting Instability of a Chord Member.

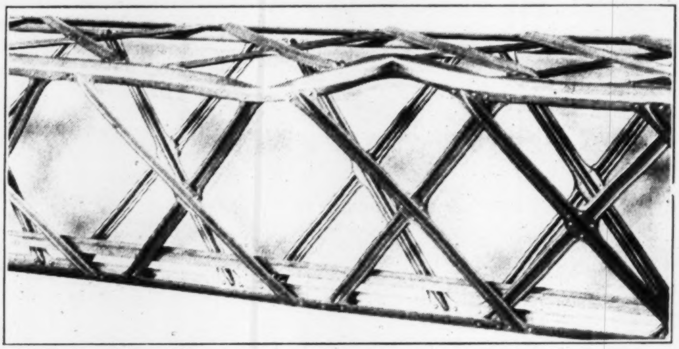


FIG. 24.—Torsional Instability of a Chord Member.

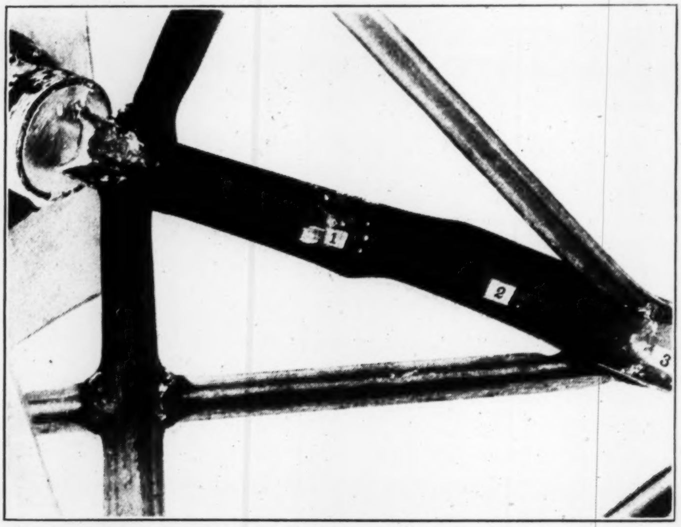


FIG. 25.—Local Instability of a Chord Member.

a cubical block may be singled out as length, another as breadth and a third as thickness, and only in exceptional cases would a distinction between them serve a useful purpose. On the other hand it is from a practical standpoint absolutely necessary to make these distinctions in the case of a long deep I beam.

Not only is this distinction between major and local instabilities arbitrary but it is frequently convenient to discuss one and the same type of instability in the same structure, now as a case of major instability and now as a case of local instability. Considering for example the structure of an airship like the Akron or the Shenandoah in its entirety as a structure, the Euler instability of one of its girders appears as a local instability and the slenderness ratios of the girders as the most important "wall thickness" ratios of the airship. Considering however the girder as a structure, its Euler instability is a major instability, and the twisting instability (Fig. 23) of one of its chord members between lattice points appears as a local instability and the slenderness ratio of the chord as its most important "wall thickness" ratio. Discussing the girder in closer detail, and considering this chord member as a structure its torsional instability (Fig. 24) in turn becomes a major instability, the crumpling and collapsing of its metal (Fig. 25) a local instability and the ratio of its thickness to its width the governing "wall thickness" ratio.

These different ways of looking at the airship structure in successively smaller parts make it possible, by studying the problem step by step, to obtain a better comprehension of it than is possible otherwise.

Returning to the simple tubular member, it is found that in determining the actual critical load for local instabilities its over-all length plays at most only a minor role, becoming of importance only when it is comparable with the linear dimensions of the section.

The over-all length of the beam or column may, however, determine whether local instability will occur, but this determination depends almost wholly upon the question whether instability or plastic deformation of the structure as a whole will or will not occur under loads too small to produce the local instability.

TYPICAL CASES OF FAILURE BY LOCAL INSTABILITY

MODULUS-DENSITY RATIO OF THE MATERIAL A LIMITING FACTOR

The characteristic factors of importance in local instability may best be seen by listing a few formulas representing some of the simpler cases in which a fairly adequate approximate theory has been worked out in detail (Fig. 26).

Two characteristic factors are common to each of these equations. The first factor is the same type of factor as appeared in the case of the

Euler column involving the reciprocal of the length and a modulus-density ratio $\frac{E_1}{d}$ or $\frac{E_2}{d}$. The modulus however is not Young's modulus but a combination of Young's modulus with Poisson's ratio. This corresponds to the fact that the stress in these particular local instabilities is highly heterogeneous (See Fig. 26), involving in each local buckle or twist its own characteristic combination pattern of shear, tension and compression. The difference in modulus although not great is of some small structural importance. It indicates a relative advantage of some 2 to 3 per cent for duralumin (Poisson's ratio 0.32) over an average steel (Poisson's ratio 0.28). The second factor is a power (different for different types of instability) of the wall thickness ratio but in contrast to the formulas for the beam, torsion member and column, in all the cases appearing in the numerator instead of in the denominator. These two characteristic factors occur in a similar manner in the formulas for every type of local instability of small portions of thin-walled structures with which I am familiar, and there are sound theoretical reasons for believing that they will occur in the same general manner in any formula which represents adequately a case of local instability, where the portions affected are small in comparison with the member as a whole.

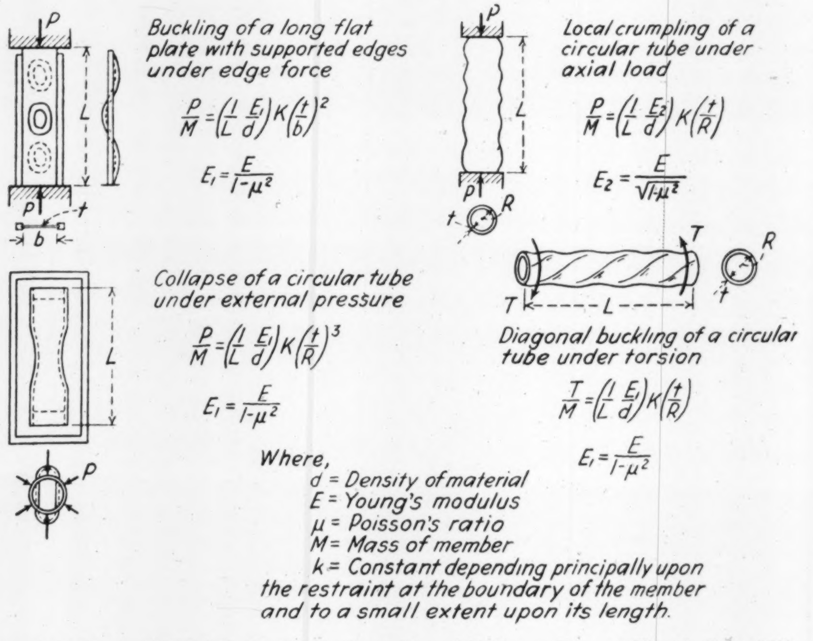

P
Buckling of a long flat plate with supported edges under edge force
$\frac{P}{M}=\left(\frac{1}{L}\frac{E_1}{d}\right)K\left(\frac{t}{b}\right)^2$
$E_1=\frac{E}{1-\mu^2}$
L
P
t
b
P
Local crumpling of a circular tube under axial load
$\frac{P}{M}=\left(\frac{1}{L}\frac{E_2}{d}\right)K\left(\frac{t}{R}\right)$
$E_2=\frac{E}{\sqrt{1-\mu^2}}$
L
P
t
R
Collapse of a circular tube under external pressure
$\frac{P}{M}=\left(\frac{1}{L}\frac{E_1}{d}\right)K\left(\frac{t}{R}\right)^3$
$E_1=\frac{E}{1-\mu^2}$
L
P
T
R
T
L
t
Diagonal buckling of a circular tube under torsion
$\frac{T}{M}=\left(\frac{1}{L}\frac{E_1}{d}\right)K\left(\frac{t}{R}\right)$
$E_1=\frac{E}{1-\mu^2}$
Where,
d = Density of material
E = Young's modulus
μ = Poisson's ratio
M = Mass of member
k = Constant depending principally upon the restraint at the boundary of the member and to a small extent upon its length.

FIG. 26.—Strength-Weight Ratio for Some Local Failures Due to Elastic Instability.

Taken at their face value these formulas state that in resisting local instability the strength-weight ratio of a structure is independent of the strength-density ratio of the material and proportional to a modulus-density ratio. Further, that for the same weight of material of the same modulus-density ratio, increase of over-all dimensions of the cross-section with decrease of its wall thickness leads to a *smaller* strength-weight ratio of the member, and consequently to a *less* economical use of the material.

THE GENERAL CASE—ALTERNATIVE TYPES OF FAILURE

We have taken up four different types of formula, each purporting to represent the strength-weight ratio of a structure under specific types of load for specific types of failure:

STRENGTH-WEIGHT RATIO OF STRUCTURAL MEMBERS WITH DIFFERENT TYPES OF FAILURE

Failure by plastic deformation under pure tension or compression

$$\frac{P}{M} = \frac{1}{L}\frac{S}{d} \quad \text{.......... Type 1}$$

Failure by plastic deformation under bending and torsion

$$\frac{P}{M} \text{ or } \left(\frac{T}{M}\right) = \left(\frac{1}{L}\frac{S}{d}\right) k \frac{A^a}{L^e} \frac{1}{(t/R)^\tau} \quad \text{.......... Type 2}$$

Failure by major elastic instability

$$\frac{P}{M} \text{ or } \left(\frac{T}{M}\right) = \left(\frac{1}{L}\frac{E_e}{d}\right) k \frac{A^a}{L^e} \frac{1}{(t/R)^{\tau_1}} \quad \text{.......... Type 3}$$

Failure by local elastic instability

$$\text{or } \frac{P}{M} = \left(\frac{1}{L}\frac{E_e}{d}\right) k \left(\frac{t}{R}\right)^{\tau_2} \quad \text{.......... Type 4}$$

$$\frac{T}{M} = \left(\frac{1}{L}\frac{E_e}{d}\right) \sqrt{A}\, k \left(\frac{t}{R}\right)^{\tau_2}$$

here;

E_e = Some particular elastic modulus

The exponents a, e, τ, τ_1 and τ_2 are all positive.

These equations present of course an enormously over-simplified picture of the problem but still contain in broad outlines the major elements which control light-weight construction. With appropriate reservations to exclude impractical freak cases, all the types of static or quasi-static loading that need to be taken into account in an engineering structure can be considered as combinations of loading each yielding equations of one of these four forms. It is worth while to discuss briefly the conclusions to be drawn from the assumption that they are an adequate presentation.

If the structure and type of loading are such that only formulas of Type 1 need be considered, all the material may be uniformly stressed to the limit of its capacity. Independent of its design its strength-weight ratio can be increased by increasing the strength-density ratio of the material. This is the case with pure tensile loads. If however the loads are compressive loads, local instability may be reached at lower loads determined by formulas of Type 4 (Fig. 27). If that is the case the strength of the structure is no longer determined by the strength-density ratio of the material but by its modulus-density ratio and its design. The material can no longer be stressed to the limit of its capacity and no increase of its strength-density ratio can increase the strength-weight ratio of the structure, in fact, the strength of the material may frequently be greatly reduced without weakening the structure in the slightest.

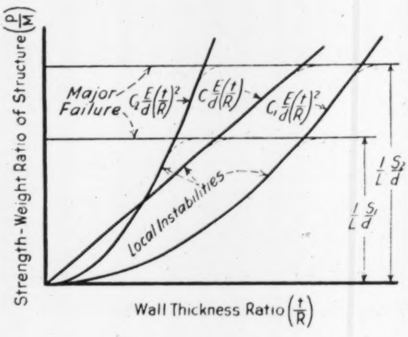


FIG. 27.—Major Failure by Plastic Yielding (Stocky Compression Member).

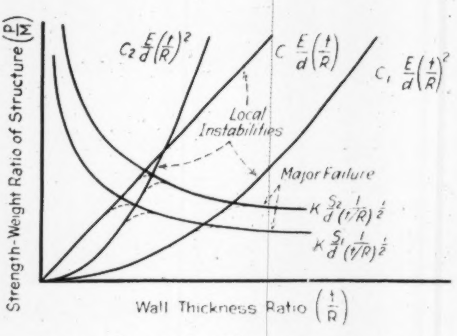


FIG. 28.—Major Failure by Plastic Yielding (Beam).

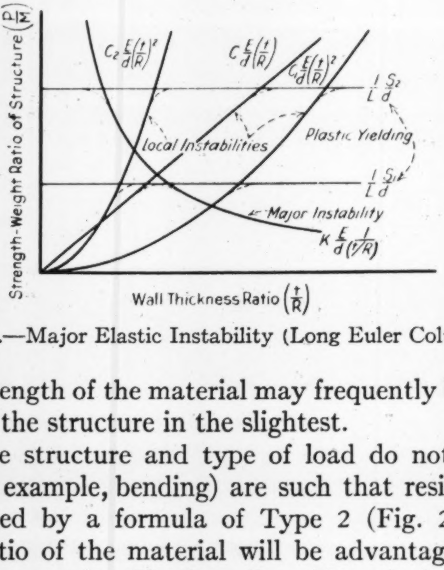


Fig. 29.—Major Elastic Instability (Long Euler Column).

Similarly if the structure and type of load do not lead to a major instability but (for example, bending) are such that resistance to a major failure is represented by a formula of Type 2 (Fig. 28) increasing the strength-density ratio of the material will be advantageous only so long as local instabilities represented by formulas of Type 4 do not occur. Again properly decreasing the wall thickness is advantageous in accordance with formulas of Type 2 only so long as it does not lead to earlier local instability in accordance with some formula of Type 4. For a given type of structure built of a material of a given strength-density ratio and a given modulus-density ratio, the theoretically most economical use of material is obtained when the wall thickness is adjusted to make major and local failure equally likely.

If the structure and load are such that failure will occur by a major instability represented by formulas of Type 3 (as in a long column) increasing the strength-density ratio of the material is of no advantage (Fig. 29). If however the wall thickness ratio is decreased, with corresponding increase of the major lateral dimensions it may happen that the strength-weight ratio of the structure will reach the limit $\frac{1}{L}\frac{S}{d}$ of plastic failure of the material before local instability represented by formulas of Type 4 occur. In this case increase of the strength-density ratio of the material will permit redesigning to secure a stronger structure. The limit however is again set by the possibility of local instability.

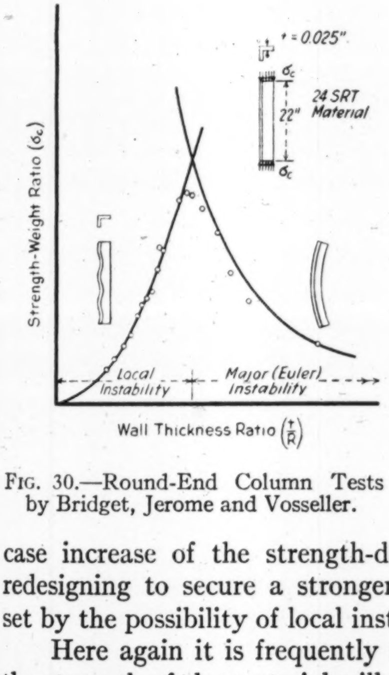


FIG. 30.—Round-End Column Tests by Bridget, Jerome and Vosseller.

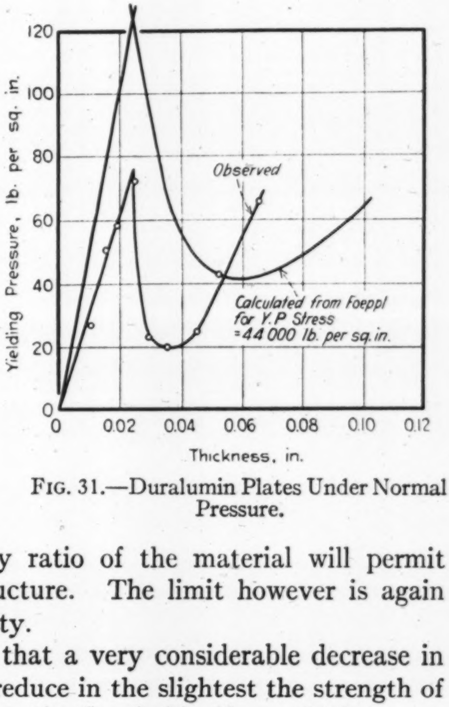


FIG. 31.—Duralumin Plates Under Normal Pressure.

Here again it is frequently true that a very considerable decrease in the strength of the material will not reduce in the slightest the strength of the structure. This was in fact the case in the airship Shenandoah.

That these very definite changes from one type of failure to another are not mere theoretical vaporings may be seen from experimental results on two widely different structures. The first[1] (Fig. 30) is a sharply demarked change with increasing wall thickness from local instability of the outstanding edges, to major instability of the structural angle acting as an Euler column. The second[2] (Fig. 31) shows an equally sharply demarked change with increasing wall thickness from plastic yielding in tension to plastic yielding in bending.

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[1] F. J. Bridget, C. C. Jerome, A. B. Vosseller, "Some New Experiments on Buckling of Thin-Wall Construction," *Transactions*, Am. Soc. Mechanical Engineers, Vol. 56, pp. 569–578 (1934).

[2] Walter Ramberg, "Flat Plates Under Normal Pressure," unpublished results obtained at National Bureau of Standards.

TRANSITION CASES AND DEVIATIONS FROM HOOKE'S LAW

Illustrations of the experimental verification of the demarcation with changing dimensions of one type of structural failure from another could be multiplied almost indefinitely, but usually the experiments show a demarcation much less sharp, one type of failure changing into the other by gradual transition as the dimensions of the structure are changed. Sometimes the gradual character of the transition shown by experiments is only apparent resulting from inadequate control of the experimental conditions, but far more often the gradual transition is real and structurally important.

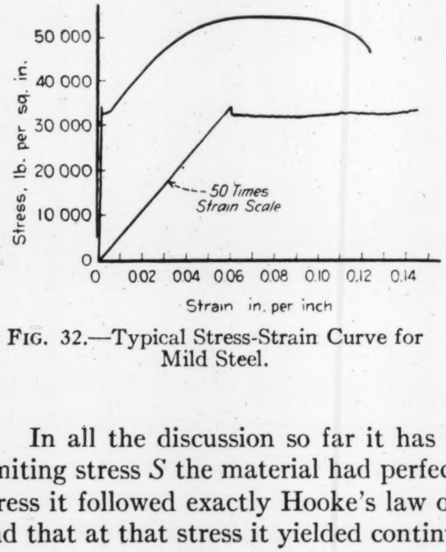

FIG. 32.—Typical Stress-Strain Curve for Mild Steel.

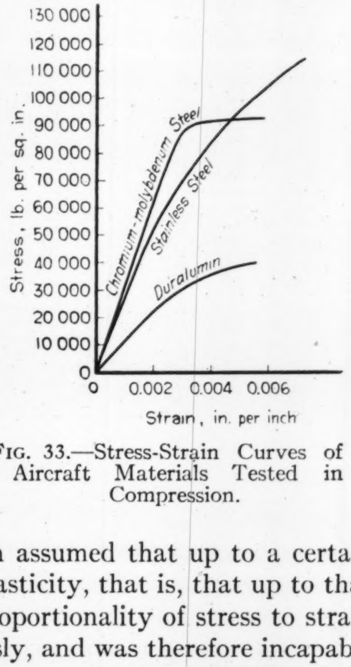

FIG. 33.—Stress-Strain Curves of Aircraft Materials Tested in Compression.

In all the discussion so far it has been assumed that up to a certain limiting stress *S* the material had perfect elasticity, that is, that up to that stress it followed exactly Hooke's law of proportionality of stress to strain and that at that stress it yielded continuously, and was therefore incapable of carrying any higher stresses. There is of course no material having these ideally simple properties. It was not so bad an assumption when the chief material in common structural use was the plain carbon structural steel (Fig. 32), but, even then the assumption was inadequate to clear up the mystery that surrounds the behavior of medium-length columns.

With the increase in the use of structural materials of markedly different stress-strain characteristics (Fig. 33), it has become more and more evident that this simple assumption can only be used as a first rough approximation, and that the details of the transition from one type of failure to another must be sought in an accurate knowledge of the stress-strain characteristics of the material.

As a result of the work of Engesser,[1] Considère,[2] Jasinski,[3] von Kármán,[4] Southwell[5] and their followers there is now a practically adequate theory of the transition between the elastic Euler instability of a long slender column and the plastic yielding under compression of a short chunk of the same material. For no other transition has a really adequate theory been worked out. Experiments and general theoretical considerations, however, show that all the transitions have certain common features which can be most easily discussed in discussing the better known case of columns.

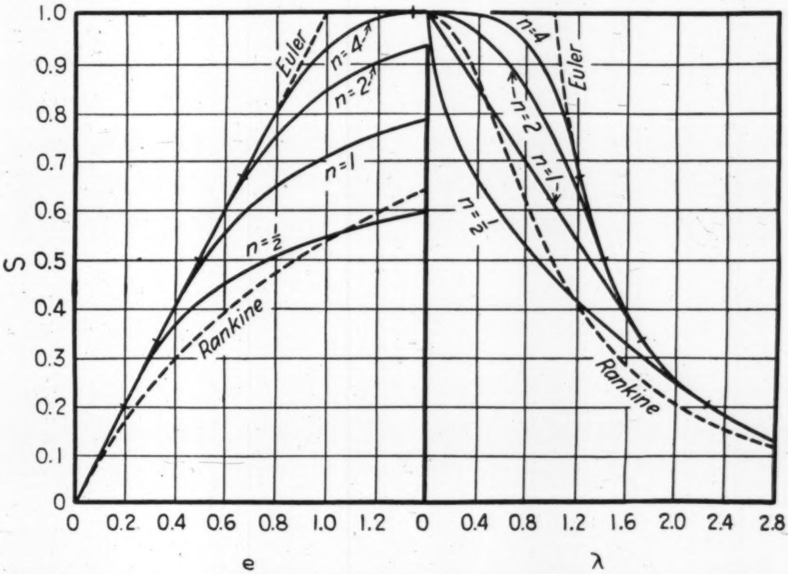

FIG. 34.—Theoretical Relations Between Shape of Stress-Strain Curve and Transition from Euler Instability to Plastic Yielding.

Under the simple assumption of Hooke's law all elastic instabilities depend upon the modulus of elasticity of the material. Without introducing the refinement of the double modulus theory of Considère which is necessary to a complete understanding of the phenomena it is readily seen that the change in slope of the stress-strain curve represents a decrease in the effective value of the modulus as the stress increases. If the dimensions of the structure are changed to raise the stress (Fig. 34)[6] at which elastic instability sets in, a more complete theory shows that the gain in strength is not so great as would be predicted by the simple theory based on Hooke's law. Instead the strength attained lies progressively farther and farther below this "theoretical" value, changing gradually into the value determined by plastic yielding alone.

---

[1] Fr. Engesser, "Zu den Knickfragen," *Schweizerische Bauzeitung*, Vol. 25, p. 88 (1895).

[2] A. Considère, "Resistance des Pièces Comprimées," Congrès International des Procédés de Construction, Paris, 1889, *Comptes Rendus*, Baudry, Paris, pp. 371–397 (1891).

[3] F. Jasinski, "Zu den Knickfragen," *Schweizerische Bauzeitung*, Vol. 25, p. 172 (1895).

[4] Th. von Kármán, "Untersuchungen über Knickfestigkeit," *Forschungsarbeiten* aus dem Gebiet des Ingenieurwesens, No. 81 (1910).

[5] R. V. Southwell, "The Strength of Struts," *Engineering* (London), Vol. 94, August, 1912, p. 249.

[6] W. R. Osgood, "Column Curves and Stress-strain Diagrams," National Bureau of Standards *Journal of Research*, Vol. 9, pp. 571–582 (1932).

Materials with markedly different shapes of stress-strain curves will show markedly different transitions between the two types of failure. Only when the stress-strain curve of the material is known can an adequate understanding of the transition be acquired.

These theoretical results have been fully verified by many series of tests on columns of widely different sizes, shapes and materials. Results obtained by Osgood[1] on specimens of chromium molybdenum and stainless (18 per cent chromium, 8 per cent nickel) steel tubes (Fig. 35) at the National Bureau of Standards are typical of these experiments. Without a knowledge of the stress-strain curve of the materials these results would seem contradictory.

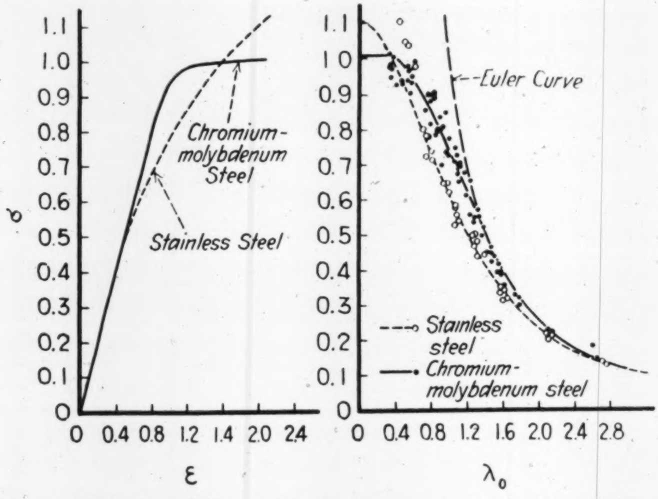

FIG. 35.—Observed Relations Between Shape of Stress-Strain Curve and Transition from Euler Instability to Plastic Yielding.

One other general characteristic of these transitions deserves special mention. When plastic yielding has become pronounced the effective value of the modulus of elasticity becomes very low, with a corresponding decrease of the resistance of the structure to elastic instability. As a consequence a structure which has failed primarily by plastic yielding, and consequently develops the full possibilities of the strength of the material, frequently deforms into the shape of a failure by elastic instability.

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[1] W. R. Osgood, "Column Strength of Tubes Elastically Restrained Against Rotation at the Ends," unpublished report of work done at the National Bureau of Standards.

Two illustrations will serve to make this point clear. Figure 36[1] shows six columns of which three show the shape typical of failure by Euler elastic instability and three the shape typical of local instability of the cover plates between rivets, yet all six columns carried as much load as could be carried by a short stocky column of the same material. All six had deformed plastically, beyond any practical usefulness, before there was any appearance of elastic instability. No redesign to prevent these instabilities could have increased the strength of the columns.

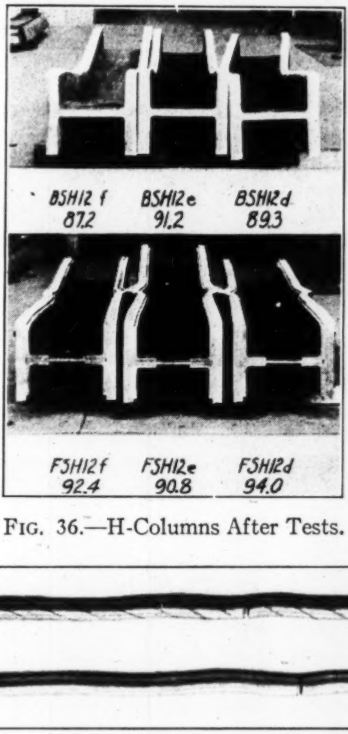

FIG. 36.—H-Columns After Tests.

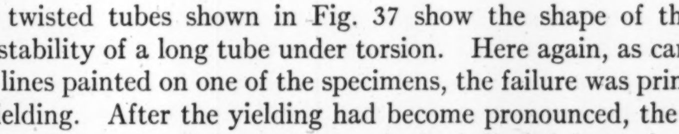

FIG. 37.—Twisted Tubes Showing Helical Elastic Instability of a Long Tube in Torsion.

The twisted tubes shown in Fig. 37 show the shape of the helical elastic instability of a long tube under torsion. Here again, as can be seen from the lines painted on one of the specimens, the failure was primarily by plastic yielding. After the yielding had become pronounced, the effective modulus was lowered sufficiently to permit the helical deformation.

These illustrations of the persistence of the appearance of elastic instability in structures where failure is primarily plastic could be multiplied indefinitely in all sorts of cases of transition between elastic instability and plastic failure.

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[1] L. B. Tuckerman and A. H. Stang, "Tests of Large Columns with H-shaped Sections," National Bureau of Standards *Technological Paper No. 328* (1926).

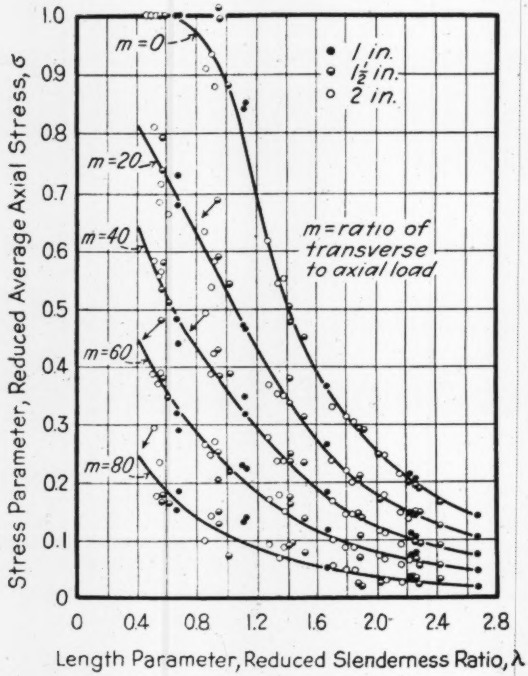

FIG. 38.—Tubing Under Varying Ratios of Axial Compression and Transverse Loading.

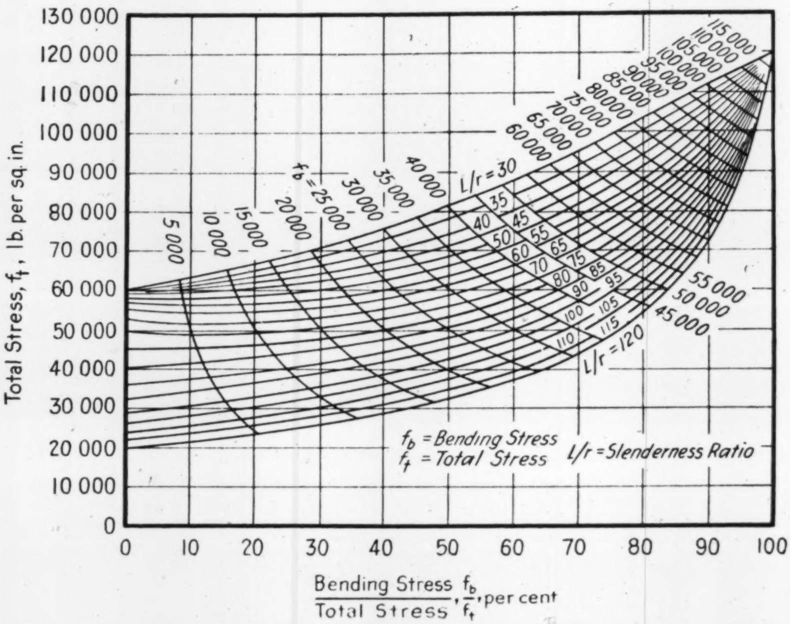

FIG. 39.—Design Chart for Tubing Under Combined Axial Compression and Tension Loading.

Only when the stress-strain curve of the material is known can these failures be properly understood. For a proper interpretation of such failures it is absolutely essential that adequate tests be made upon coupons cut from the material from which the structures were made. The conclusions drawn from many structural tests have been absolutely valueless, or of very doubtful value, because of a failure to correlate them with adequate coupon tests.

Even more complicated than these cases of transition are the phenomena under varying types of loading. The tubular members in a welded fuselage are not only subjected to axial loads, tension or compression, but to bending moments applied at their welded joints, and in addition not infrequently to lateral loads distributed along their length. The tubular axes of ailerons, elevators and rudders must carry varying combinations of torsional and bending loads. These are merely simple illustrations. In general the designer of aircraft must take into account the strength of the structure and its component parts under all sorts of loadings combined in all proportions.

The case of major failure of tubing under varying ratios of axial compression and transverse bending is a simple illustration (Fig. 38). The test data here presented are converted into a design chart (Fig. 39).[1] Here the lower limit of the chart and the lower portion of its left-hand side are fixed by the modulus-density ratio of the material and no increase in the strength of the material can be of any use. For all the rest of the chart, the strength-density ratio enters in varying degrees, which are fairly well determined by simple formulas from values read from the curves. The chart, however, is valid only for tubes whose wall thickness ratio (t/R) is so great that local crumpling does not occur. If that were to be included it would be necessary to add a third coordinate and change the lines in the chart to surfaces, which slope down to zero for very thin walls.

OPTIMUM USE OF MATERIAL

However great the complications, the structural problem of lightweight construction retains the same basic character. It is the problem of securing the best possible balance between plastic yielding, dependent upon the strength-density ratio of the material, and major and local elastic instabilities dependent upon its modulus-density ratio, and finally the transitions between these, dependent upon an accurate knowledge of its stress-strain curve (Fig. 7).

LIGHT *Versus* HEAVY MATERIALS

One further limitation of the formulas needs to be mentioned. Taken at their face value they indicate that in all cases, structures of the same weight, equally strong in all respects, can be made of materials of different density provided their modulus-density and strength-density ratios are the same. This is only approximately true. The formulas of Type 4 purport to represent the conditions under which local elastic instability will occur. These formulas are all based on the assumption of very thin walls (Fig. 8)—walls so thin that the wall thickness is a negligible quantity when added to or subtracted from the major lateral dimensions of the section. Of two such structures the one made of the lighter material will have the thicker walls.

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[1] L. B. Tuckerman, S. N. Petrenko, C. D. Johnson, "Strength of Tubing Under Combined Axial and Transverse Loading," National Advisory Committee for Aeronautics, *Technical Note No. 307* (1929).

No really satisfactory theory has been worked out for any local instability in thicker walls where this approximation ceases to give an adequate representation. We know however from theoretical considerations, as well as from tests, the sign of the error involved. In all cases the wall with the greater thickness will have somewhat greater stability. Where the use of the heavier material would require extremely thin walls there is a very certain advantage, but of uncertain and variable magnitude, in the use of thicker walls of lighter materials, in some cases even with a somewhat smaller modulus-density ratio.

DESIGN FACTORS

The four types of formulas[1] express explicitly the effect of over-all dimensions, wall thickness, and the properties of the material.

In the constants k, $k_1 \ldots\ldots k_2$ which have been only casually mentioned lie concealed all the possibilities of improvement by details of design. Especially in the case of local instabilities represented by formulas of Type 4, these constants may be changed enormously by relatively small changes in shape. A simple illustration will help to make this clear. A flat steel ribbon is a very flimsy object, but when given a slight permanent longitudinal curvature it acquires the remarkable stiffness of the familiar metal rule.

In searching out the particular geometrical shapes that will give the largest values of these constants, and consequently yield the highest resistance to failure of thin walls by local instability, theory has been of help only in giving the broadest of guidance. One general principle may be stated: "So far as possible in thin-sheet construction avoid large unsupported areas, either plane or with large radii of curvature." This principle was known empirically long before it had been given any theoretical background. Corrugated sheet and corrugated lattices, bulb angles, lipped flanges, dished edges of lightening holes, and many other illustrations have long been familiar, and theory has merely confirmed common knowledge.

Even this principle has its exceptions. Before Wagner,[2] it had been felt that elastic instability must be avoided at all cost. Wagner had the genius to recognize that certain types of instability of a part of a structure might be safely permitted, and, far from being a detriment would in appropriate cases lead to still lighter safe construction than had previously been achieved. These are instabilities which occur under small deformations and are self limited under larger deformations. The shear wrinkles (Fig. 40) of Wagner beams still cause uneasiness in the minds of many pilots and passengers but experience is teaching them to forget their fears. The limits of the possibilities of this type of structures with self limited instability are yet to be explored. The chord members of Wagner beams must of course be amply safe against instability and to them the general principle still applies.

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[1] See p. 27.

[2] H. Wagner, "Ebene Blechwandtraeger mit sehr duennem Steg Blech," *Zeitschrift für Flugtechnik und Motorluftschiffahrt*, Vol. 20, pp. 200, 227, 256, 279, 306 (1929).

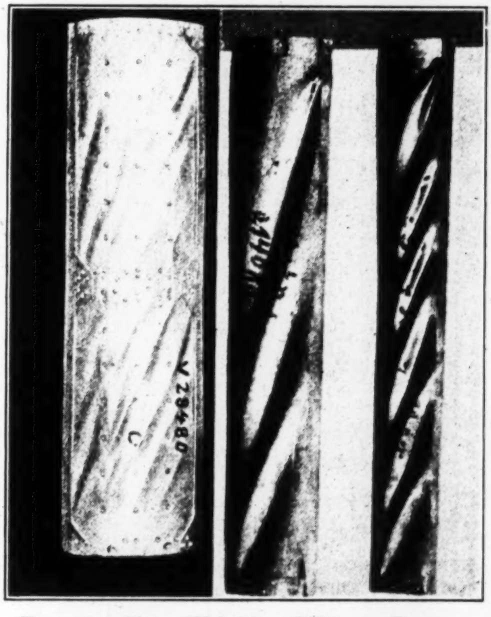

FIG. 40.—Shear Wrinkles of Wagner Beams.

The actual application of the principle of avoiding large unsupported flat areas still must be almost wholly empirical, and brilliant empiricism has resulted in marked improvements in design. In airship girders, for example (Fig. 41), successive improvements in design to improve the resistance to local instabilities have raised the average stress from a range of 16,000 to 24,000 lb. per sq. in. in the girders of the Shenandoah to a range of from 32,000 to 40,000 lb. per sq. in. in the girders of the Akron and Macon.

It is here that materials and structural testing have played their greatest role in the rapid development of light-weight structures.

THE ROLE OF STRUCTURAL TESTING

If it had been necessary to build a complete airplane or a complete airship and try it out in service to determine the value of any suggested change in detailed design, progress would have been immeasurably slower. Fortunately the general theory of structures makes it possible in many cases to determine, with reasonable adequacy, the forces acting upon elements of the structure small enough to test in the laboratory. Hundreds and thousands of such structural elements have been tested out in aircraft factories, until by empiricism, checked by tests, more and more efficient designs have been produced.

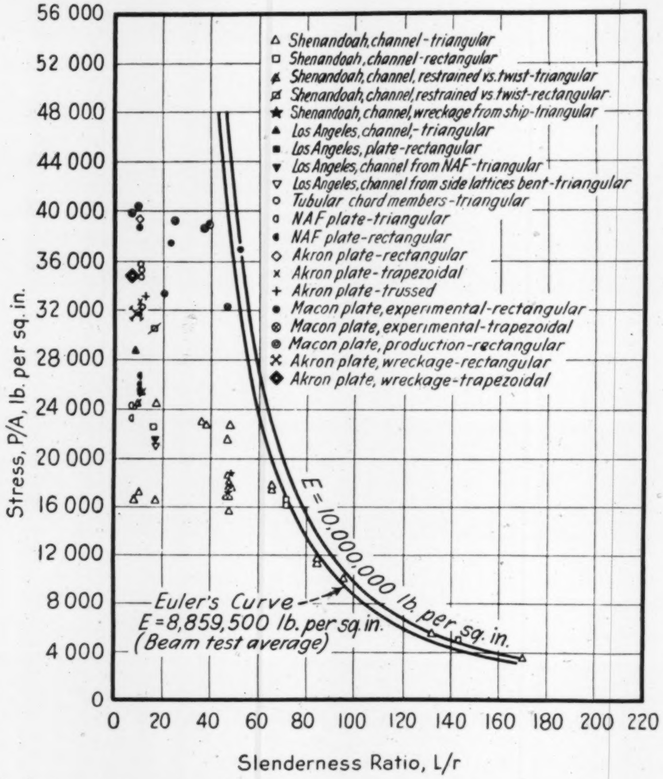

FIG. 41.—Column Tests on Airship Girders.

Unfortunately most of these tests have been planned merely to answer specific questions about specific details of construction. In the great majority of tests the designer has been content to test the structure without parallel coupon tests of the material so that the validity of the results is limited to structures built of that particular material.

If men knew fully the mathematical relationship between the detailed geometrical shape of a structure, the properties of the material of which it was constructed, and its strength to withstand the types of loading to which it would be subjected, it would, in thought at least, be possible to dispense with structural tests, and draw conclusions from materials tests alone. However, even if that goal were reached, men have not infinite mathematical knowledge nor infinitely rapid computing machines. Suggest to any mathematician that he obtain a solution of a non-linear differential equation of the second order which has prescribed values over the surfaces of such complicated shapes as are common in light-weight structures. He will tell you that even for linear differential equations, and such simple boundaries as spheres, cylinders, cones, parallelopipeds, etc., the solutions are extremely complicated, and that it is utterly fantastic to dream that men will ever master so complicated a problem as you suggest, by mathematical computations alone.

Well-planned structural tests accompanied by adequate materials tests furnish the only answer. Isolated tests on single structures ordinarily have little permanent value. To yield results of permanent value, the tests must be systematic series of tests on typical structural elements covering ranges of materials, over-all dimensions, wall thickness and types of loading, sufficiently wide to determine all the types of instability and plastic yielding likely to occur in practical designs, determining their limiting conditions and the effect of the stress-strain curve of the materials upon the transitions between them.

Because of such systematic series of tests we are no longer in ignorance of the conditions controlling the major failure of columns. Because of such systematic series of tests we are beginning to draw safe inferences about some few types of local instability, putting empirically determined constants into equations of the types we have discussed.

Many more such systematic series of tests are needed if light-weight construction is to realize its greatest possibilities.

OTHER MATERIAL FACTORS

In judging a material of construction other factors have to be weighed, in addition to the density, the modulus of elasticity, and a measure of static strength determined in some way from its stress-strain curve, but for the most part these other factors do not enter quantitatively into design computations. Minimum elongation is customarily specified, not because of any definitely known relation between the elongation of the material and the strength of the structure but because experience has shown that too low elongation makes the material difficult or impossible to fabricate and in some obscure way seems to be associated with lessened security against shock loads. Sometimes impact tests are specified in the hope of a more direct assurance of safety in case of shock. Similarly a minimum reduction of area is sometimes specified

Resistance to corrosion is another factor which must ever be considered. Here the original strength of the structure is not involved but merely its ability, either bare or with suitable protective coatings, to retain that strength when exposed to the deteriorating influences of moisture and the other corrosive agents found in the air. For most, though not all, of the materials used, a reasonable resistance to corrosion has been achieved.

It would be delightful if material could be wished into its final structural shape. Since this cannot be done, the possibility and practicability of forming the material into desired shapes and fastening it together by such methods as bolting, riveting, crimping, soldering, brazing and welding must be considered. This is frequently a deciding factor in the choice of materials and has been a subject of much study. Nevertheless from the standpoint of the ultimate structural possibilities of a material these are secondary considerations. Improvements in the technique of shaping and fastening metals are continually making possible wider uses of materials with a consequent improvement in design.

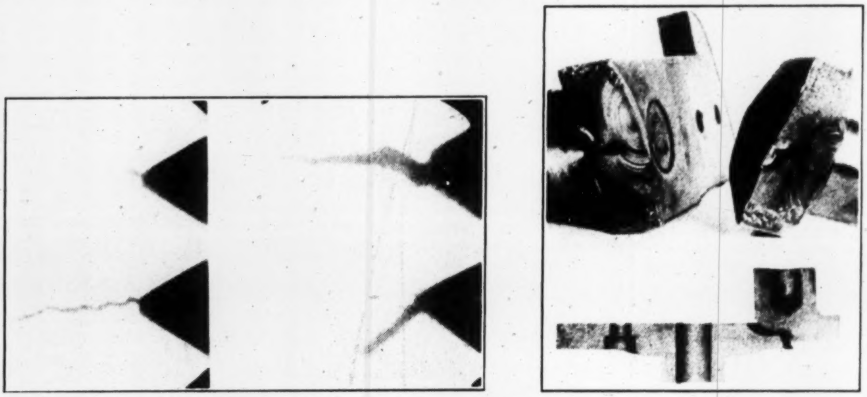

FIG. 42.—Illustrating Dangers of Reentrant Angles and Rapid Changes in Section.

In some few cases it is possible to use the measures of the resistance of a material to fatigue in quantitative calculations of the strength of a structure. We need to know much more about the ability of materials to stand up under fluctuating stresses, and the time may come when calculations based on this property of the material will be a part of any thorough-going design computation. For the present, however, the crying need in the prevention of fatigue failure is a keener appreciation of the mandatory nature of the commandment which should be written large in the decalogue of every designer of moving structures: "Thou shalt not build structures which are subject to high fluctuating stresses, with rough surfaces, sharp reentrant angles (Fig. 42), rapid changes in section or thin or rough outstanding flanges." The failure to heed this commandment has been and continues to be the cause of many disastrous failures, not infrequently resulting in the loss of human life. Records show the failure of a propeller blade because the maker proudly stamped his name and patent numbers on it.

COMPARISON OF AVAILABLE MATERIALS

In the brief survey of materials that follows only the density, modulus of elasticity and static strength will be considered.

For many years engineers used the ultimate tensile strength of the material as the sole measure of the strength of a material and it still is an important measure. With further knowledge of the plastic deformation of materials the old "yield point" by drop of beam was added, and for many years there was argument whether "tensile strength" or "yield point" should be used as a basis for design. When materials of markedly different stress-strain characteristics came into common use (Fig. 33) which showed no "yield point" a demand arose for some other measure of the resistance to plastic deformation. There has been and still is much discussion as to the best measure to use. The discussion of the relation between column strength and stress-strain curve makes it clear that no one measure will serve all needs.

TABLE II.—COMPARATIVE STRUCTURAL PROPERTIES OF SELECTED MATERIALS.

Material	Density, lb. per cu. in.	Young's Modulus, 10^6 lb. per sq. in.	Yield Strength, 10^3 lb. per sq. in.[a]	Tensile Strength, 10^3 lb. per sq. in.	Young's Modulus-Density Ratio, 10^6 in.	Yield Strength-Density Ratio, 10^3 in.	Tensile Strength-Density Ratio, 10^3 in.
Silk fabric	0.0120[b]	....	....	8.7[b]	....	....	720
Cotton fabric	0.0193[b]	....	....	8.6[b]	....	....	446
Manila rope	0.0283-0.0340	....	....	8.4- 13.5	....	....	289- 424
Wood	0.0156-0.0197	1.3- 1.7	5[c]- 7[c]	9.4[d]- 11.5[d]	83- 86	321[c]-355[c]	584[d]- 603[d]
Magnesium alloys	0.065	6.5	....	34 - 43	100	....	523- 662
Beryllium	0.067	27.4	....	....	409	....	....
Aluminum alloys	0.101	10.4-10.6	30- 58.3	55 - 68	103-105	297-583	545- 680
High-strength steel wire	0.283	27.7-28.6	178-251	260 -298	98-101	629-887	919-1053
Structural steel	0.283	29.0-29.4	33- 57.6	58.2- 97.5	102-104	117-204	206- 345
Carbon-steel tubing	0.282	27.6	36- 57.4	55 - 75.3	98	128-204	195- 267
Chromium-molybdenum steel tubing	0.282	29.7-30.3	75-167	95 -178	105-107	266-594	337- 633
Stainless steel tubing	0.285	25.8	135-149	175 -186	91	474-523	614- 653
Beryllium-copper alloys	0.295 -0.298	15.2-19.0	....	59 -196	51- 64	....	198- 664
Monel metal	0.318	25 -26	25[e]- 60[e]	70 - 90	79- 82	79-189	220- 283

[a] Stress corresponding to a strain of 0.002 greater than elastic strain computed for this stress unless otherwise noted.
[b] Thickness determined under pressure of 0.25 lb. per sq. in.
[c] Value for compressive strength.
[d] Value for modulus of rupture.
[e] Stress corresponding to strain of 0.005 in.

However, for the purpose of materials specification some definite measured departure from the ideal straight line of Hooke's law sometimes different for different classes of material and for different uses of the same material, has proven fairly adequate. Further, when properly chosen, it has been possible to use such a single measure as the basis for many design computations. The values given in Table II as "yield strength" are values determined in accordance with current specifications. For woods, compressive strength and modulus of rupture have been taken as most nearly comparable to yield strength and tensile strength. Examination of Table II shows that when both values are available the relative values for the different materials fall in practically the same order whether $\frac{\text{yield strength}}{\text{density}}$ or $\frac{\text{tensile strength}}{\text{density}}$ is used as a measure of the strength-density ratios of the material, so that for our purpose it is only necessary to discuss the latter.

The materials listed range in density from 0.012 lb. per cu. in. for silk fabric to 0.318 lb. per cu. in. for monel metal, a ratio of over 26 to 1, their tensile strengths range from 8400 lb. per sq. in. to nearly 300,000 lb. per sq. in., a ratio of over 35 to 1, their moduli of elasticity from 1,300,000 to 30,000,000 lb. per sq. in., a ratio of over 23 to 1.

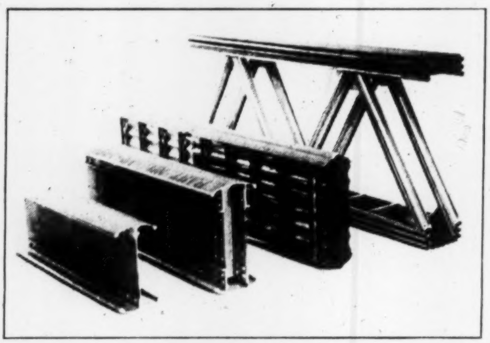

FIG. 43.—Aluminum-Alloy and Alloy-Steel Beam Sections.

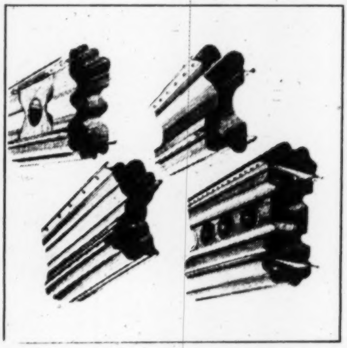

FIG. 44.—Alloy-Steel Beam Sections.

In contrast to these wide variations, their modulus-density ratios (with the one remarkable exception of beryllium) range only from 51 to 107 (for comparative purposes the particular unit used does not matter, but if we wish to be specific we might, with apologies to the purists, say 51 to 107 mega inches) a ratio only slightly over 2 to 1.

Excluding the beryllium copper alloys, which because of low modulus-density ratio, and monel metal which, in addition, because of low strength-density ratio, are definitely less well suited for use as main structural elements in light-weight construction, the modulus-density ratio ranges only from 83 for wood to 107 for chromium-molybdenum steel tubing. Limiting the comparison to the structural metals the range is only from 91 for stainless (18 per cent chromium, 8 per cent nickel) steel to 100 for aluminum alloys and 107 for heat-treated alloy steels similar to chromium-molybdenum steels.

Allowing for the 2 to 3 per cent advantage of aluminum alloys on account of Poisson's ratio and the certain though indefinite advantage of thicker walls, it is seen, especially for small structures where the walls are necessarily very thin, that there is a certain, though small, advantage of the aluminum alloys over the steels in resisting local instabilities.

For larger structures with thicker walls, the higher modulus-density ratio of the chromium-molybdenum (and similar heat-treated alloy steels) may outweigh this advantage and in some cases probably does. In addition fairly satisfactory welding processes are already available for these steels, and no equally satisfactory process is at present available for the aluminum alloys. The advantages of aluminum alloys and alloy steels are so evenly balanced that the designer is often hard put to it to decide between the two. It seems to me probable that the choice is frequently decided by the designer's familiarity with one or the other of the two classes of materials.

TABLE III.—DENSITY, MODULUS-DENSITY RATIO, AND STRENGTH-DENSITY RATIO OF SELECTED MATERIALS.

Material	Density, lb. per cu. in.	Young's Modulus-Density Ratio, 10^6 in.	Tensile Strength-Density Ratio, 10^3 in.
Silk fabric	0.0120[a]	....	720
Cotton fabric	0.0193[a]	....	446
Manila rope	0.0283–0.0340	....	289– 424
Wood	0.0156–0.0197	83– 86	584[b]– 603[b]
Magnesium alloys	0.065	100	523– 662
Beryllium	0.067	409	....
Aluminum alloys	0.101	103–105	545– 680
High-strength steel wire	0.283	98–101	919–1053
Structural steel	0.283	102–104	206– 345
Carbon-steel tubing	0.282	98	195– 267
Chromium-molybdenum steel tubing	0.282	105–107	337– 633
Stainless steel tubing	0.285	91	614– 653
Beryllium-copper alloys	0.295 –0.298	51– 64	198– 664
Monel metal	0.318	79– 82	220– 283

[a] Thickness determined under pressure of 0.25 lb. per sq. in.

[b] Value for modulus of rupture.

A designer accustomed to design aluminum alloy structures (Fig. 43) might not feel as much confidence in his ability to master the radically different designs necessary to secure the same strength-weight ratio from alloy steels. Some of these (Fig. 44) would seem very bizarre to one not familiar with the structural problems involved. Conversely a designer used to alloy steels might feel more at home with them.

Unless the elastic characteristics of the stainless (18 and 8) steels can be considerably improved, their low modulus-weight ratio (91) will always tend to hinder their wider use. Their resistance to corrosion will, however, in many cases outweigh the 14 to 16 per cent disadvantage of their modulus-weight ratio.

Aside from the difficulty in protecting them from corrosion, the magnesium alloys because of their slightly lower modulus-density ratio (100) offer little promise of seriously competing with aluminum alloys or alloy steels in main structural elements where local failure controls the design. Their future is more promising in relatively bulky parts or fittings not subject to local failure, where mere volume or perhaps other non-mechanical considerations are of importance.

These comparisons based upon density and modulus-density ratios can be made with considerable confidence and a reasonable expectation that no technical developments at present to be expected will materially alter the conclusions. Density and modulus of elasticity are stubborn properties of the material. The most violent differences in heat treatment and mechanical working, differences in treatment which change the strength of the material by ratios as great or even greater than 10 to 1, can at most cause a change of a few per cent in either modulus or density.

No such confidence can be placed on comparison based on the strength-density ratios (Table III). At the time the first airplane flew no strong light alloys comparable to duralumin were known, and although some high-strength alloy steels were known they were not commercially available in the wealth of forms, tube, sheet, and rolled shapes now at the disposal of the aircraft constructor.

High-strength steel wires with the remarkable strength-density ratios of 1000 (in units of 1000 in.) were of course to be had, but the highest strength ratios available in carbon steel, sheet, tubing or small rolled shapes was of the order of 250, while rope, fabric and woods, were available with strength-weight ratios ranging from around 400 to 700. The relatively low modulus-weight ratio of woods (83 to 86) was no real disadvantage since the low density of wood (less than one fifth that of duralumin) made possible wall thicknesses so great as to exclude in large measure the possibility of local instability. With the added advantage of its relatively low cost and the ease with which wood could be shaped it was natural that the first aircraft should be built of wood and wire. No other equally strong, light-weight construction was available.

The discovery of high-strength aluminum alloys and the technical developments in the production of alloy steels has made possible the rapid advance in light-weight metal construction. For members subjected to purely tensile stresses, steel wire still continues to have a marked advantage over any other material. For columns, beams, girders and stressed skin, from the strength-density standpoint, there is now little difference between the 662 of magnesium alloys, 653 of the alloy steels, and the 680 of aluminum alloys. In fact if the data in Table III had been gathered from other sources these values might have appeared in any other order.

FUTURE POSSIBILITIES

It would be rash to make any definite predictions concerning future developments. Great progress has been made in the past twenty years in producing stronger and ever stronger metals, and there are physicists and metallurgists who bring forward arguments which seem to prove that the advance so far made falls far short of what we may expect, when we have learned to make full use of interatomic forces.

The great progress in aircraft construction brought about by the discovery of the strong light alloys, and the remarkable strength possibilities of the alloy steels might lead one to hope for still greater progress in the future. However, for the present at least, further advance in the development of stronger alloys offers but slight promise of radically lighter aircraft construction. With the strength-density ratios now available it is possible to make full use of the strength of the material only in a small portion of the structure of an aircraft. Local instability, governed by the modulus-density ratio, still sets the limit, and with the materials now available any considerable progress can only be made by improvement in design.

I do not wish to leave the impression that this is a pessimistic conclusion. We are certain that systematic materials and structural tests such as are now being carried on in a number of laboratories, combined with improvements in the technique of forming metals and fastening them together, will lead to aircraft markedly superior in strength and lightness. If and when these improvements in design and fabrication are carried far enough to eliminate local instabilities, the strength-density ratio of the material will again be the limiting material property. Then and only then will stronger materials give greater strength for lighter weight. When that time arrives the designer in redesigning to reach the utmost of his possibilities will again be limited by the reappearance of local instability.

BERYLLIUM—A DREAM OF POSSIBILITIES

The importance of the modulus-density ratio in determining the strength of aircraft explains why some designers look with hungry eyes on the unique value for beryllium among the modulus-density ratios. With material of such enormous relative stiffness available in a workable, structurally useful form the picture would be radically altered.

All the beryllium I have seen was as brittle as glass. Some metallurgists have contended that brittleness was inherent in its crystal structure, and that ductile beryllium was an impossibility. Some metallurgists hoped that as an alloying element it might infect magnesium, aluminum, copper, steel or what not with some useful measure of its high modulus of elasticity. From this investigation have come useful beryllium copper alloys (Table III) but no alloys of high modulus-density ratio.

At laboratories in England and America, by elaborate processes, small pieces of ductile beryllium have been produced. For years it was thought that drawn tungsten wire was an impossibility, but now it is a common commercial product. Will there be a similar development of ductile beryllium? If so will it have a strength-weight ratio comparable with or greater than the aluminum alloys and the alloy steels?

Not so long ago "pure" beryllium, that is, as pure as available, cost several hundred dollars a pound. How much cheaper can it become? If it becomes available in workable form, how low a price must it reach to be economically practical in aircraft construction?

All these are tantalizing dreams.

CONCLUSION

Whether or not these tantalizing dreams are realized, we shall, with better designs, better and more easily fabricated materials, and improved technique in fabrication, build safer, stronger and lighter aircraft. We shall feel confident of their safety because it will be based on more and more adequate theory, and more and more knowledge of the strength of structures drawn from materials and structural tests planned in the light of theory.

I can do no better in closing than quote the specifications for a perfectly designed structure written long before modern aircraft had become a reality. Chaises are not aircraft, but the principles involved are the same:

> Now in building chaises, I'll tell you what,
> There is always somewhere a weakest spot,—
> In hub, tire, felloe, in spring or thill,
> In panel, or cross bar, or floor, or sill,
> In screw, bolt, thorough-brace,—lurking still,
> Find it somewhere you must and will,—
> Above or below or within or without,—
> And that's the reason, beyond a doubt,
> That a chaise *breaks down*, but *doesn't wear out*.
>
> But the Deacon swore (as Deacons do,
> With an "I dew vum," or an "I tell yeou,")
> He would build one shay to beat the taown
> 'n' the keounty 'n' all the kentry raoun';
> It should be so built that it *couldn'* break daoun:
> "Fur" said the Deacon " 't's mighty plain
> Thut the weakes' place mus' stan' the strain;
> 'n' the way t' fix it uz I maintain,
> Is only jest
> T' make that place uz strong uz the rest."

We shall never reach this goal in aircraft, but we can strive to approach it.

SYMPOSIUM ON SPECTROGRAPHIC ANALYSIS

SPECTROSCOPIC ANALYSIS OF STEELS—THE INFLUENCE OF NON-HOMOGENEOUS SAMPLES

By W. R. Brode[1]

In the evaluation of a quantitative spectrographic method of analysis it is essential to have available a set of known or reference samples whose composition is definitely known. These standard samples may be prepared by mixtures of known amounts of elements or compounds or by careful chemical analysis of a number of samples of varying composition. Such a standard series should contain a reasonable number of samples and should be well distributed within the concentration range under study so as to provide a sufficient number of points on the calibration curves.

Through the cooperation of Mr. J. D. Sullivan, analyst for the Battelle Memorial Institute, a series of analyzed samples has been made available for our study. For a period of years Sullivan has been making a careful study of the residual metals in open-hearth steels[2] (Fig. 1). The object of this study has been to determine the trend in composition changes brought about by the use of scrap materials. The elements studied included copper, tin, nickel, chromium and manganese. The samples were obtained from 18 producers representing different geographic centers, and are representative of the major portion of open-hearth steel produced in the period studied.

The composition average for each year was determined from the average of the values of the individual manufacturers supplying samples, and it was these latter samples which were studied spectrographically. The ranges in composition of a portion of the 1934 samples are shown in part in Table I. It will be noted that in some cases there is a considerable concentration variation. Calibration curves covering these ranges should be particularly useful in developing the application of spectrographic analysis to this type of steel.

Through a misunderstanding we were under the impression at the start of this study that the samples were homogeneous in character, that is, with the turnings or drillings obtained from a melt which had been made up of samples removed from each melt produced. It was soon discovered after the plates were prepared on some 18 samples and readings of line intensities started, that the observed values fluctuated considerably beyond the allowable error expected in the spectrographic method (5 to 10 per cent of the amount observed). Inquiry confirmed a suspicion aroused by this variation, namely, that the yearly company sample was a composite sample of the turnings taken from the samples of each melt, but that there had been no melting of the composite samples to produce homogeneous samples. Our photographs, even though exposures were as long as 2 min., represented for the most part separate samples wherein the composition might well be quite different from that of the averages.

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[1] Associate Professor of Chemistry, Ohio State University, Columbus, Ohio.

[2] J. D. Sullivan and C. E. Williams, "Residual Metals in Open-Hearth Steels," *Metals and Alloys*, Vol. 3, p 240 (1932); Vol. 4, p. 151 (1933); Vol. 5, p. 145 (1934); Vol. 6, p. 134 (1935).

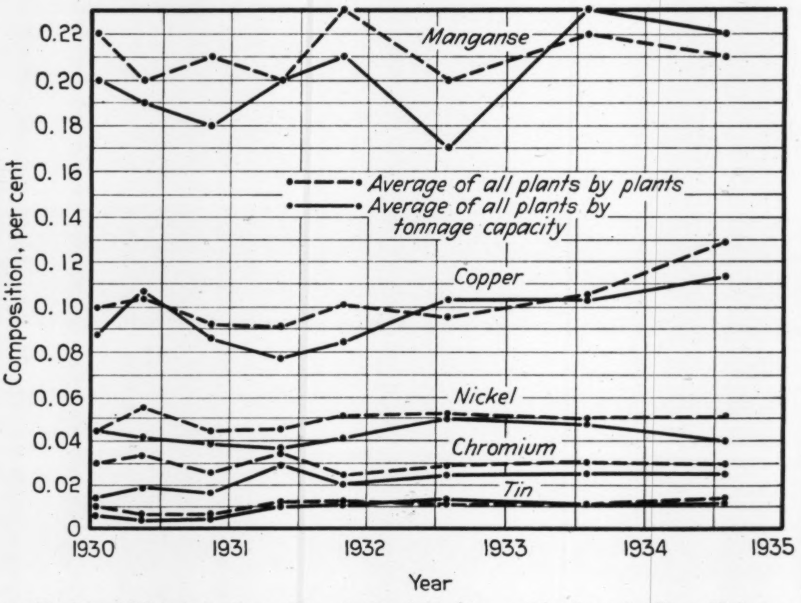

FIG. 1.—Residual Metals in Open-Hearth Steel (Sullivan, *Metals and Alloys*, Vol. 5, p. 145 (1935).

In spite of this disturbing turn in the problem it was felt worth while to complete the measurement of the plates so as to obtain some definite data on the influence of homogeneous character in spectrographic samples.

The general procedure in the analysis involved the production of an arc between a carbon electrode (negative) and the turnings (positive), the latter being held in a nickel-iron cup (capacity about 5 cc.). As a part of the original plan it had been hoped that a comparison might be made of the log-sector and density-photometry methods—hence a departure was made from the usual methods of illumination in placing the condensing lens adjacent to the sector and that in turn adjacent to the slit. This results in the reduction of intensity but at the same time provides for better sampling through longer exposures.

TABLE I.—RESIDUAL METALS IN OPEN-HEARTH STEELS.

SAMPLE	NICKEL, PER CENT	COPPER, PER CENT	TIN, PER CENT	MANGANESE, PER CENT	CHROMIUM, PER CENT
No. 1	0.115+	0.058	0.007	0.25	0.038
No. 2	0.040	0.196	0.006	0.10+	0.029
No. 3	0.074	0.318+	0.041+	0.13	0.036
No. 4	0.091	0.218	0.024	0.18	0.039+
No. 5	0.039	0.135	0.023	0.20	0.037
No. 6	0.038+	0.083	0.006	0.22	0.020+
No. 7	0.086	0.281	0.025	0.17	0.037
No. 8	0.075	0.150	0.015	0.25	0.034
No. 9	0.039	0.049+	0.005+	0.40+	0.035
No. 10	0.044	0.075	0.010	0.27	0.030
No. 11	0.049	0.078	0.008	0.27	0.031

TABLE II.—LOG-SECTOR VALUES OF A SINGLE SAMPLE.

	3246 Å Iron	3247 Å Copper	3262 Å Tin	3268 Å Iron	3271 Å Iron	3273 Å Copper	3276 Å Iron	3414 Å Nickel	3415 Å Iron	3492 Å Nickel	3495 Å Iron	3515 Å Nickel	3516 A Iron
	6.8	7.2	4.8	4.8	7.8	8.1	4.0	6.1	6.0	5.5	6.8	5.5	5.2
	6.5	7.5	4.8	4.5	7.5	8.0	4.2	6.5	6.0	6.2	7.3	6.8	5.5
	6.0	7.0	4.0	4.0	7.2	7.5	4.0	5.3	5.0	5.0	6.0	5.5	4.8
	5.5	6.5	4.2	3.8	6.5	6.8	2.8	4.5	4.0	4.0	5.7	4.5	3.8
Sum	24.8	28.2	17.8	17.1	29.0	30.4	15.0	22.4	21.0	20.7	25.8	22.3	19.3
Difference from average sum of all iron lines.........		+1.7	−8.7			+3.9		−4.1		−5.8		−4.2	
Difference from sum for adjacent iron lines.........		+3.4	+0.7			+1.4							

	4009 Å Iron	4014 Å Iron	4021 Å Iron	4030 Å Manganese	4033 Å Manganese	4034 Å Manganese	4245 Å Iron	4247 Å Iron	4254 Å Chromium	4274 Å Chromium	4289 Å Chromium	4352 Å Iron
	8.5	8.4	9.2	11.0	10.3	10.0	5.8	7.8	8.5	8.0	7.8	8.8
	8.2	8.8	9.0	11.2	10.5	10.3	7.5	8.5	9.5	9.7	8.8	9.0
	8.5	8.5	8.6	10.5	10.2	10.0	7.0	8.8	8.8	8.5	7.8	9.0
	7.5	7.2	7.0	9.0	8.8	8.5	5.5	7.8	7.2	6.8	5.8	7.5
Sum.........	32.7	32.9	33.8	41.7	39.8	38.8	25.8	32.9	34.0	33.0	30.2	34.3
Difference from average sum of all iron lines.........................				+15.2	+13.3	+12.3			+7.5	+6.5	+3.7	

Grand total of all iron lines........... 344.4
Average sum of all iron lines.......... 26.5

TABLE III.—RELATIVE DENSITY VALUES OF A SINGLE SAMPLE.

	3246 Å Iron	3247 Å Copper	3262 Å Tin	3268 Å Iron	3271 Å Iron	3273 Å Copper	3414 A Nickel	3415 Å Iron	3492 Å Nickel	3495 Å Iron	3515 Å Nickel	3516 Å Iron
	22.5	10.3			17.0	10.4	25.0	25.8	26.0	21.5	24.0	25.3
	14.6	9.8	19.2	23	11.5	9.5	19.4	19.3	20.8	13.9	18.0	19.0
	8.8	6.6	14.6	17.8	7.7	6.4	10.4	11.6	12.7	8.4	11.6	12.0
	13.0	10.8	19.5	21	9.6	10.2	17.1	17.5	18.4	12.4	16.5	17.5
	14.0	12.2	18.7	20.9	9.6	11.8	16.3	17.7	18.1	12.4	16.4	17.5
	23	17.0	25.5	27	17.0	16.0	22.5	24.0	23.6	19.6	22.6	23.9
	20.7	15.5	25.2	26.8	16.6	15.2	23.0	23.8	24.3	19.6	22.6	23.6
Sum.........	116.6	82.2	122.7	136.5	89.0	79.5	133.7	139.7	143.9	107.8	131.7	138.8
Ratio........	1.41		1.11		1.13		1.04		0.75		1.05	

	4009 A Iron	4014 Å Iron	4021 Å Iron	4030 Å Manganese	4033 Å Manganese	4034 Å Manganese	4245 A Iron	4352 Å Iron	4247 Å Iron	4254 Å Chromium	4274 Å Chromium	4289 Å Chromium
	16.5	17.0	15.6	19.2	24.2	25.9	22.8	16.0	15.6	22.2	20.4	24.4
	8.3	9.4	8.3	5.2	5.7	6.1	12.9	7.4	7.9	6.0	6.4	7.2
	5.5	6.2	5.7	4.3	4.6	4.8	8.6	5.4	5.9	5.1	5.4	5.8
	7.8	9.3	7.7	5.0	5.5	6.1	12.0	7.1	7.7	6.4	6.8	7.9
	7.8	8.5	7.7	5.3	5.5	5.9	12.2	7.3	7.8	6.9	7.0	8.4
	12.5	14.0	12.5	8.0	8.7	10.6	18.0	11.1	11.7	11.6	12.6	14.5
	12.3	13.6	12.2	7.9	8.5	9.6	17.3	10.5	11.5	9.8	10.4	13.3
Sum........	70.7	78.0	69.7	54.9	62.7	69.0	103.5	64.6	68.1	68.0	69.0	81.6
Total..........	218.4			186.6			236.2			218.6		
Ratio..........................	1.17						1.08					

In general four exposures by the log-sector method and seven by the normal method of exposure were made on each sample. The photographs were taken on an E 316 Hilger Spectrograph (3-m. focal length), the spectral range used was that of 3200 to 4400 Å for the usual 10-in. plate (Fig. 2).

Since the use of added internal standards was not practical, a group of iron lines was chosen for internal comparative standards. In the case of copper the principal lines 3247 and 3273 Å were compared with iron lines 3246 and 3271. The iron lines were of nearly the same intensity as the copper lines under comparison.

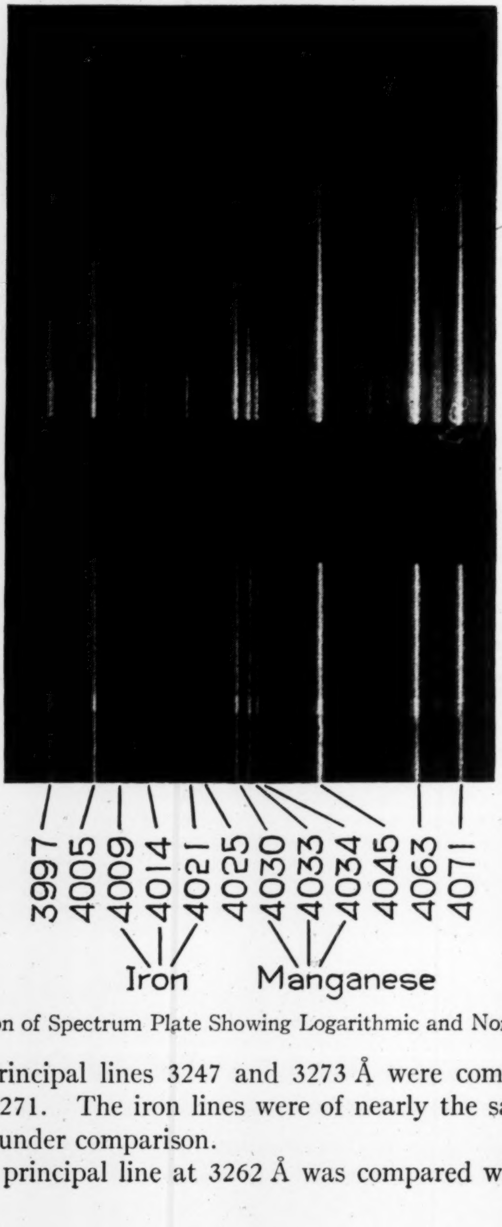

FIG. 2.—Portion of Spectrum Plate Showing Logarithmic and Normal Spectrum.

For tin the principal line at 3262 Å was compared with the iron line at 3268 Å.

For nickel the principal lines at 3414, 3492 and 3515 Å were compared with iron lines at 3415, 3495 and 3516 Å.

For manganese the principal triplet lines at 4030, 4033, 4034 Å were compared with the iron lines at 4009, 4014 and 4021 Å.

For chromium the principal lines at 4254, 4274, 4289 Å were compared with the iron lines at 4245, 4247 and 4352 Å.

In part of the tabular representation of data, especially when the lines to be compared did not differ greatly in intensity from the average of the iron lines, the mean value for 13 iron lines was used as a basis of comparison. In certain cases, such as copper or tin, where the observed lines were quite different in intensity from the average iron line, the adjacent iron lines of similar intensity were used. The sums of all readings for each line were used in place of averages, since they are proportional.

In the determination of the log-sector values (Table II), the lengths of the lines were measured with the Bausch & Lomb measuring magnifier plate with scale. The scale was divided in 0.1 mm. and an estimation to 0.02 mm. is possible—although actually the error in locating the ends of the lines is much larger, that is, 0.1 to 0.2 mm.[1]

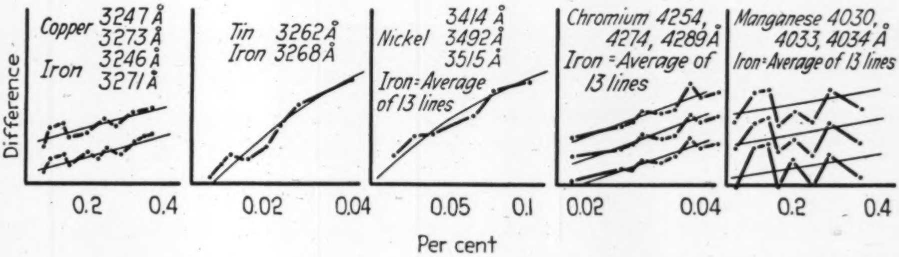

FIG. 3.—Difference in Line Intensity as Determined by the Logarithmic-Sector Method.

The determination of intensity values (Table III) was made on the Bausch & Lomb density comparator—using a Weston photronic cell and Leeds & Northrup Type H. S. galvanometer (a total swing of 32 cm. from light to dark). The ratio of deflection values gave a measure of relative line intensity.

The graphical representation of the data, Fig. 3, as has already been mentioned, amply demonstrates the necessity for homogeneous samples. In addition it gives some indication as to the range of variation which might be expected in the component portions of the sample—for example, it would appear that tin does not fluctuate as much as copper.

By either method the analysis of non-homogeneous samples gives unsatisfactory results. In order to demonstrate that the fluctuations were due to lack of homogeneity in the sample, a series of observations was made by the solution method on five of these samples (Nos. 1, 2, 3, 6 and 9). In each case the series of photographs was quite uniform and the intensities varied directly with relative concentration of the metal. Since the solution method involved the use of a second carbon and a salt rather than metal turnings, we did not feel justified in examining the entire series by this method and preparing calibration curves.

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[1] L. C. Martin, S. A. Burke and E. G. Knowles, *Transactions*, Farady Soc., Vol. 31, p. 495 (1935).

The indications are that the results from this method will be highly satisfactory. The ease of handling the turnings in the arc method makes this method quite desirable and it is hoped that homogeneous samples will shortly be available which will permit the preparation of a series of calibration curves for this method.

DISCUSSION

Mr. C. C. Nitchie.[1]—There is one question I should like to ask Mr. Brode. It is a point that has been brought out in many published papers. What indications are there of the necessity of choosing for comparison lines of the same spectral type? That is, comparing arc lines with arc lines, or spark lines with spark lines? Is there any indication to show that that is an essential consideration in a method of this type?

Mr. W. R. Brode.[2]—I am not certain that it is an essential consideration, although we carefully chose arc lines for comparison with arc lines and we tried to choose lines which represented the same type of ionization.

Mr. Nitchie.—The question of sampling is also important. In spectrographic analysis we are using only a very minute amount of material for our sample and it is highly essential to know that that sample is definitely representative of the large quantity of the material it is supposed to represent. The standard methods being developed by Committee E-2 on Spectrographic Analysis will include some restrictions as to the method of sampling to be employed in order to get thoroughly reliable results.

Mr. E. J. Welker.[3]—What is the average weight of the samples used in this examination?

Mr. Brode.—About five grams was used in the case of turnings. A more preferable sample is a $\frac{1}{4}$-in. steel rod. The sample need not be more than $\frac{1}{2}$ in. in length. In the case of a steel rod, one should make both the upper and lower parts of the same material. In the case of a sample of turnings, we used a pure carbon rod for the upper electrode. The cyanogen bands produced by the carbon did not interfere to any appreciable extent with the observations. In the case of an analysis on the inside of a piece of boiler tube to determine whether there was any lead present, we used a pure iron rod for the upper electrode, using the unknown tube section for the lower. The lines of any impurity elements came out very well since the iron lines did not interfere with the additional lines.

Mr. A. J. Phillips.[4]—I should like to know how large a sample was consumed and what guarantee there is that the consumption of the sample meant an equal consumption of each element under investigation. In other words, if there were zinc in the sample, was there any guarantee that the zinc line would not have been much more intense than was representative of the zinc content?

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[2] Associate Professor of Chemistry, Ohio State University, Columbus, Ohio.
[3] Amplex Manufacturing Co., Division of Chrysler Corp., Detroit, Mich.
[4] Superintendent of Research, American Smelting and Refining Co., Maurer, N. J.

MR. BRODE.—There is no guarantee that the line would not be more intense; although if you start with a known sample and use a standard calibration curve, you can reproduce your results, although the actual intensity of the zinc line might be greater than you would expect for a given amount of zinc. If you used that zinc line as a standard and took another sample of similar composition, you would get similar intensities. Referring to the amount of the sample actually consumed, one may use a few milligrams in a determination; although the longer exposures will consume larger amounts of sample. However, that brings up the question that too long exposure may result in the removal of certain volatile elements entirely, so that the length of exposure should be controlled. If one is devising a standard method of analysis, there should be a definite time of exposure, so as to insure uniform sampling.

MR. D. H. REYNOLDS.[1]—I should like to ask a question about the technique of using drillings or lathe turnings as samples; how soon after the start of the arc was the exposure made?

MR. BRODE.—The arc was usually burned for at least a minute before the exposure was made.

MR. C. E. MARGERUM.[2]—I should like to express a little difference of opinion on the matter of sampling, especially in the matter of burning the arc long enough to acquire a constant spectrum. We should have the average of the cross-section covered by the crater, and I believe by that method, having arced the sample long enough for the spectrum to become constant, we should then have a true representation. Another thing; is it possible to determine carbon in this manner?

MR. BRODE.—With regard to the sample, I too, much prefer a steel rod to turnings. With regard to the analysis of carbon itself—I believe that Mr. Emery, of Cleveland, has worked in that particular field and I think he is of the opinion that it can be done. I have found it very difficult. The analysis that he carried out was in the presence of argon or helium gas, in which he determined the intensity of the carbon line in the neighborhood of 2400 Å. Carbon, oxygen and such elements cannot be determined with any ease; at least in steel.

MR. W. E. BUCK.[3]—I should like to ask Mr. Brode if he sees any hope of spectrographic analysis becoming a useful tool for rapid qualitative analysis for the detection of odd elements in steel, such as alloy contamination, so that the steel may be diverted in case the contamination is objectionable for the quality for which the heat was originally charged.

MR. BRODE.—I think it is eminently suited for qualitative analysis, even better than for quantitative analysis. A person who has worked with the spectroscope for some time will learn to recognize in a photograph the lines of pure iron. Two spectra, one of pure iron, the other of steel, can be compared at once and in a few minutes one can tell all the elements present which are not iron and even make some estimate as to the amount.

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[2] Material Engineer, U. S. Navy, Bureau of Ordnance, Navy Dept., Washington, D. C.
[3] Metallurgist, Granite City Steel Co., Granite City, Ill.

MR. L. S. FLETCHER.[1]—I wonder if Mr. Brode can give us some idea how elaborate the apparatus must be to make a qualitative steel analysis?

MR. BRODE.—It must be a good deal more elaborate for the qualitative analysis of ferrous materials than for non-ferrous materials. I think that, for qualitative steel analysis, one should not be satisfied with anything less than 10-in. dispersion for the range from 2000 Å to 4000 Å. With regard to the spectrograph itself, almost all concerns that make spectrographs produce instruments of that type at about $1500. I think the smaller instruments are not suitable for steel analysis because one must be able to get in between the iron lines to observe the lines of the other elements. I think that the best apparatus for steel analysis is the Littrow, meter and a half, which costs around $3000.

MR. EUGENE WAINER.[2]—In making the intensity-concentration curves, it appears that, wherever possible, the ultimate lines of the elements under consideration were used. Do you find that the slope of the analytical curve varies with the spectral type of line?

MR. BRODE.—The slope of the curve varies with the type of line.

MR. THOMAS A. WRIGHT.[3]—Mr. Brode has brought out a very interesting and vital point in reference to the heterogeneous results obtained due to the fact that the sample was a composite of a number of others. This matter of the preparation and integrity of a sample should be kept particularly in mind in spectrographic work. Small samples are used because larger ones are often not available or because of electrode limitations. For these and other reasons, it is not always feasible to overcome segregation by solution and aliquoting of the sample.

A piece of material as sampled may have appeared homogeneous and to all intents and purposes have been so as a mass, but in the very operation of sampling and drilling, one often tends to dissociate into soft and hard constituents. I have seen this particularly noticeable in a high-silicon aluminum alloy where millings taken over the cross section rather checked each other, but when, in an endeavor to prove the results, these millings were ground, there was at times a tremendous difference in composition between the finer, harder or brittle particles and the coarser, softer particles. Copper-lead and types of alloys in which we have both hard and soft and tough components must be handled with this in mind.

It is something which causes us considerable concern at times and we have sometimes found it advisable actually to screen and weigh the coarse and fine particles, choosing some suitable mesh which would give us the best line of demarcation.

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[2] Spectroscopist, Titanium Alloy Manufacturing Co., Niagara Falls, N. Y.

[3] Technical Director, Lucius Pitkin, Inc., New York City.

At times, these may be recombined in their proper proportion if sufficient sample can be taken into solution before arcing or sparking, but if the material is to be exposed in the solid form, then it may be advisable to treat the coarse and fines separately and recalculate to the original basis.

An analytical result is usually nothing but an opinion expressed in figures, whether these are derived by chemical analysis, by spectrographic analysis or a combination of the two. If the procedure chosen is the proper one and the technique employed of sufficiently high grade, then the figure reported represents, within the degree of precision required, a portion of the sample. It does not necessarily follow, however, that the portion weighed or measured out represents the sample nor that the sample truly represents the specimen or lot of material in question though much depends upon the purpose for which the analysis is required.

Too much emphasis cannot be placed on proper drawing of a sample and proper preparation. Even those appreciative of the physical and mathematical phases of sampling sometimes overlook the fact that chemical changes may and often do occur, particularly in the drying or preparation stages, with age and because of atmospheric conditions.

The laboratory that employs data obtained from single determinations may not appreciate the importance of the subject but the one that runs analyses in duplicate or more no doubt will.

Finally it should not be necessary to emphasize the advisability of running all spectra in duplicate, but apparently that point too is often overlooked.

MR. R. A. WOLFE.[1]—In making intensity measurements, how was the effect of background from the carbon bands overcome?

MR. BRODE.—The carbon bands did not occur in the region of the chosen lines with the possible exception of one of the nickel lines. The other lines were not in the region of the carbon bands. No attempt was made to eliminate them. The amount was very small, if any was present, and the general method of taking a large number of iron lines over the entire region I think to some extent eliminated that effect.

MR. R. K. KNIGHT.[2]—What are the possibilities of a very rapid spectrographic analysis, such as is encountered in steel mill control work?

MR. BRODE.—I have made no attempt to work out a rapid method. I should think that the sample could be cut and arced, unless one preferred to arc with a carbon against the sample itself without cutting. The time of exposure, say, making several photographs, would probably run up to two or three minutes. Development will take about six minutes and the negative may be read wet, if necessary, or it may be dried by immersion in alcohol. I think the analysis could be made within a period of fifteen minutes without much difficulty.

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[2] Chemist, Materials Testing, Chrysler Corp., Detroit, Mich.

THE USE OF THE SPECTROGRAPH IN THE PLATINUM INDUSTRY

By H. E. Stauss[1]

The method of use of the spectrograph in the platinum industry has been largely determined by two fundamental factors, the value of the platinum metals and the chemical inertness of the metals. The high value of platinum makes small lots of metal important commercially and leads to the careful recovery of all forms of scrap from all sources. The purchase of scrap from any source in turn requires the maintenance of an assay laboratory ready to analyze any material sent in for refining. This assay laboratory is available for special analysis to anyone in our company and has largely made routine quantitative spectrographic analyses unnecessary. The chemical inertness of the platinum group metals has made infeasible the development for spectrographic analysis of methods requiring solutions. The inability to use standard solutions means that quantitative analysis requires standard alloys. The obvious difficulties with maintaining an inventory of standard alloys of the precious metals of sufficiently fine gradation of composition to be useful have acted as a deterrent upon the adoption of quantitative spectrographic analysis and have led to confining analyses to those of a qualitative or a semi-quantitative nature, that is, to analyses in which the order of the amount of impurity is determined. The uses of the spectrograph have been, therefore, in those fields in which it was superior because of its speed, its thoroughness, or its modest demands for samples. They fall roughly into three categories: (1) identification of alloys, (2) special analyses for minor impurities, and (3) control of the purity of platinum and its alloys for special purposes.

Identification of Alloys:

Two of the important characteristics of the platinum industry are the large number of alloys in use, most of them of minor importance, and the many users to whom platinum and all its alloys are indiscriminately "platinum." From time to time it is necessary to duplicate a sample of metal called "platinum" that is obviously not platinum. Because of the time and cost involved, such a sample frequently cannot be submitted to chemical analysis, and instead, an attempt is made to identify the alloy from its use and its physical properties, such as thermal electromotive force, resistance, or hardness. Where such tests are not possible, or are not conclusive, spectrographic analysis is used. Spectrographic analysis and the measurement of some physical property usually are sufficient to identify standard alloys.

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Special Analyses for Minor Impurities:

The demand for special qualitative analyses for minor impurities comes from two sources. There is the metal returned by the trade because of unsatisfactory service. This is examined for contaminations. There is also the eternal question within the company of minor impurities, a question which becomes acute when nearly unworkable alloys are being made. So far as returned ware, largely laboratory ware or thermocouples, is concerned, the difficulties usually arise from overdoing a good thing. Platinum and its alloys are inert to many chemical reactions and to many combinations of chemical reactions and high temperatures where other metals fail. In many cases they are used with compounds of metals and metalloids which are safe, while the metals and metalloids themselves are dangerous because they form relatively low fusing alloys with platinum. In such use reducing conditions occasionally occur accidentally and a low-melting alloy is formed. When this type of failure cannot be definitely recognized by the user, spectrographic analysis of the area of failure is made to determine whether contamination has occurred. Arsenic, silicon, and phosphorus have been found to cause the most trouble with laboratory ware. In much the same way thermocouples are occasionally contaminated under reducing conditions.

The second need for qualitative analyses arises within the company. The refining of the platinum group metals is difficult; many of the alloys used are very difficult to work; many of the products are subject to close specifications in thermal electromotive force, hardness, or resistance; and the high temperatures of melting and annealing make the question of contamination during working very real. When trouble does occur, it is very difficult to establish the exact origin, and the spectrograph has won a place as an independent judge in such cases. Either it readily establishes and identifies contaminations or it disproves the theory of contamination. Moreover, it usually is able to locate the origin of contamination. It has been called upon to test platinum, rhodium, and iridium sponges, and to examine metal after nearly every stage of manufacture. The importance of this use can hardly be overestimated because it plays so large a role in guiding refining and manufacturing operations.

Control of the Purity of Platinum and Its Alloys for Special Purposes:

The third category of use of the spectrograph, the control of purity, is probably the most important. The most prominent instance of this use is the control of the gauze catalyst used in the oxidation of ammonia to nitric acid. The spectrograph serves to control the purity of the finished gauze and later, by examining the returned used gauze, it determines the contaminations accumulated during operation as a catalyst. The earlier stages of manufacture are controlled by routine physical tests, including thermal electromotive force and tensile strength measurements. Finally when the roll of gauze is all completed, a sample is taken from one end for spectrographic analysis. It must be absolutely free of impurities that could be picked up in annealing, working, or weaving. The returned gauzes are examined for any unusual impurities that may have been picked up during use. To one familiar with the history of the use of platinum and of its alloys with rhodium for the oxidation of ammonia, the value of such examinations is obvious. With erratic activation of the catalysts, with possible poisons in the gases, and with apparently a thousand other harmful factors constantly present as bugbears to haunt the operator of an ammonia oxidation plant, spectrographic control of the gauze catalyst before and after use has played a leading role in developing the present-day smooth-running operation of the oxidation plant.

In all of the uses of the spectrograph as applied to platinum, the importance of qualitative analysis has been stressed. Some of the natural urge towards quantitative analysis has been weakened by the special conditions in the industry—the maintenance of an assay laboratory and the difficulties of a method of quantitative analysis that would be sufficiently convenient and thorough to be of value. What is required is a method which would give the amounts of any and every impurity in any and every alloy with a limited number of exposures. However, there is a growing need for the quantitative analysis of minor impurities and it is planned to investigate this field. The limitations in the platinum industry—the insolubility of the samples, the varied physical character of the samples, and the expense of carrying any considerable inventory of standard alloys—all point to the use of the logarithmic sector with the arc, and as soon as opportunity permits, the use of the sector will be investigated. For qualitative analysis the arc has been adopted with a bead of metal on graphite electrodes as the sample. This is found to be the most satisfactory method of excitation, and apparently to be more sensitive than the spark. The beads are readily made, whereas standard-size electrodes for spark analyses are often impossible to obtain. Even standard alloys for semi-quantitative analyses are readily made directly by melting several components in the arc to form a bead of the desired alloy. Semi-quantitative analyses made by the bead and arc method appear to give satisfactory results. The unknown spectrum is compared either with standard spectra on other plates or with standard spectra on the same plate, using beads of the same weight, usually 50 mg., and equal, short exposure times of 20 sec. for both the known and the unknown samples. It is hoped that with the logarithmic sector it will be possible to build up a collection of curves for quantitative analysis such as have been made, for example, by Hitchen for tin,[1] that will enable most platinum alloys to be analyzed readily and without intercomparison with standard alloys.

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[1] C. S. Hitchen, "Quantitative Estimation of the Impurities in Tin by Means of the Quartz Spectrograph," *Technical Publication No. 494*, Am. Inst. Mining and Metallurgical Engrs. (1933).

QUANTITATIVE SPECTROGRAPHIC ANALYSIS OF MAGNESIUM ALLOYS FOR MANGANESE AND SILICON

By J. S. Owens[1] and T. M. Hess[1]

Introduction

Spectrographic analysis has been used at The Dow Chemical Co. since 1925. It has found considerable application in the general control of the processes and products of a large, diversified chemical manufacturing plant. Its applications have increased, due to experience and general development of technique, from purely qualitative analysis and from the visual estimation of small amounts of impurities in various materials to accurate quantitative analyses, which have in several cases replaced chemical methods.

The basis of quantitative spectrographic analysis lies in the correlation between the intensities of the spectral lines of the element under test and the concentration of that element in the specimen. One of the greatest difficulties confronting this type of analysis has been the accurate and convenient conversion of the density of the spectral line on the photographic plate into the intensity of that line in the light source. While various methods of conversion cited in the literature, for example the use of the logarithmic spiral disk, have been tested in the course of our work, the one found to be the most successful is that using the Hansen step-diaphragm.

Throughout most of our work the densities of the spectral lines have been measured with a Gaertner microdensitometer, but recently a Hilger non-recording microphotometer has been installed and is now under test. The use of this latter instrument, it is believed, will increase the rapidity and the precision of the analyses. Moreover, the personal factor in the measurement of density should be greatly reduced by its use.

Another step which has greatly aided in increasing the accuracy of our analyses has been the proper choice and standardization of the light sources appropriate for the excitation of the spectra of different materials to be analyzed.

The present paper describes, not a new method of analysis unknown in the literature, but a successful industrial application of spectrographic methods, made possible by careful control of each step of the technique.

A fruitful field in our plant for quantitative spectrographic analysis, and the one in which our major efforts in this phase have been expended, is that of the control of the development and manufacture of magnesium alloys known as Dowmetal alloys. These are magnesium-base alloys containing approximately 90 per cent magnesium together with varying amounts of manganese, silicon, aluminum, zinc, copper, iron, cadmium, and tin either as alloying constituents or as impurities. The necessity of a frequent check on several or on all of these constituents in the alloy manufacture makes this problem particularly susceptible to attack by the spectrographic method. Since the information necessary for the estimation of the concentrations of several elements may be obtained from a single spectrum, this method of analysis possesses the advantages of rapidity and of economy of sample. The accuracy yielded by the two being comparable, spectrographic methods have, to a considerable extent, replaced chemical methods for the analyses of these alloys.

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In the present paper the general method of analysis is described, using manganese and silicon as specific examples. Routine analyses have been developed for manganese in the range from 0.009 per cent to 1.5 per cent, and for silicon in the range from 0.005 per cent to 2.0 per cent, to a degree of precision and of accuracy equal to that of routine chemical analysis.

ANALYTICAL TECHNIQUE

The method of analysis used is based upon the relationship existing between the concentration of a constituent of the specimen and the intensities of its spectral lines in the light source. This relationship is experimentally determined from the relative intensity of a selected pair of spectral lines, one of the element under test and the other of the "internal standard"(**2**)[1] element. The relative intensity of the pair of lines is obtained from their measured densities by means of a density-logarithm of intensity calibration pattern placed upon each plate(**1,3,4,5,6,7,9**).

Excitation of the Spectrum:

A condensed spark discharge between solid alloy electrodes is used for the excitation of the spectrum. The mechanical and electrical conditions of the spark are controlled in accordance with a predetermined constant condition. The length of the spark gap is maintained at 5 mm. between electrodes of 11 by 3 mm. cross-section. A 1-kva., 110 to 25,000-v. Thordarson transformer furnishes the spark voltage. In series with the spark gap is an air-core inductance of 0.00003 henry; in parallel with the gap is a capacitance of 0.00577 μf.

The condensed spark, under these controlled conditions, was found to yield more accurate analyses of magnesium alloys than an arc between solid electrodes. Even when held in massive, water-cooled holders, the burning of the electrodes prevents the maintenance of an arc of constant spectral characteristics.

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[1] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 67.

The spectra are recorded on Eastman Polychrome plates by means of a Bausch & Lomb medium quartz spectrograph. A slit width of 0.06 mm. is used. No lens is used between the spark source and the slit. The source is placed at a sufficient distance from the slit to insure that all the light from the source which passes through the slit will also pass through the spectrograph lens.

It was found that the maintenance of the spark discharge for a minimum time interval of 6 min. before exposure greatly increased the accuracy of the analysis.

Choice of Spectral Lines:

The spectral lines chosen for use in the analysis should fulfill the following conditions:

1. The chosen lines should have suitable densities over the ranges of the elements under test occurring in the analyses and should lie in a wave length region of constant, or of only slightly varying, contrast of the photographic plate.
2. Whenever possible only lines of the same character should be compared; that is, arc lines with arc lines, and spark lines with spark lines.

The spectral lines chosen were:

Magnesium: 2915.5 Å and 3074.1 Å (both spark lines)
Manganese: 2605.7 Å, 2610.2 Å, and 2949.2 Å (all spark lines)
Silicon: 2516.1 Å and 2881.6 Å (both arc lines)

An analysis may be made by the use of one pair of lines, one of magnesium and the other of the element under test. The use of another pair of lines serves as a check of the analysis.

Determination of the Analytical Curves:

On each plate, in addition to the spectra of the specimens, is placed a density-logarithm of intensity calibration pattern. This is done as follows: the slit of the spectrograph is opened to its maximum width, a Hansen(3,6,9) step-diaphragm (which becomes effectively a stepped-slit) is inserted directly before the slit, and the continuous spectrum of an appropriate light source (condensed spark of aluminum in a hydrogen atmosphere(9)) is photographed. Since the widths of the steps of the diaphragm vary in a known way (logarithmically to the base 1.5), this photograph produces on the plate a pattern consisting of a series of bands of known variations of intensity.

The densities of a pair of selected lines, one of the element under test and the other of the internal standard (magnesium, the major constituent of the alloy), and of the steps of the intensity calibration pattern, at appropriate wave lengths between 2400 Å and 3800 Å, are measured with a Gaertner microdensitometer. From the densities of the steps of the calibration pattern is obtained the density-logarithm of intensity calibration curve of the plate(4,5,6). The logarithms of the relative intensities of the pairs of spectral lines are then obtained by applying their measured densities to this curve.

This procedure is carried out for a series of alloys of known composition in which the concentration of the element under test varies over the range required for the analyses. From the data so obtained an analytical curve(8,10,11) relating the logarithm of the relative intensity to the percentage of the element under test in the alloy is drawn. These analytical curves, once determined, are used for all future analyses. Figure 1 shows representative analytical curves for the analysis of Dowmetal alloys for manganese and silicon.

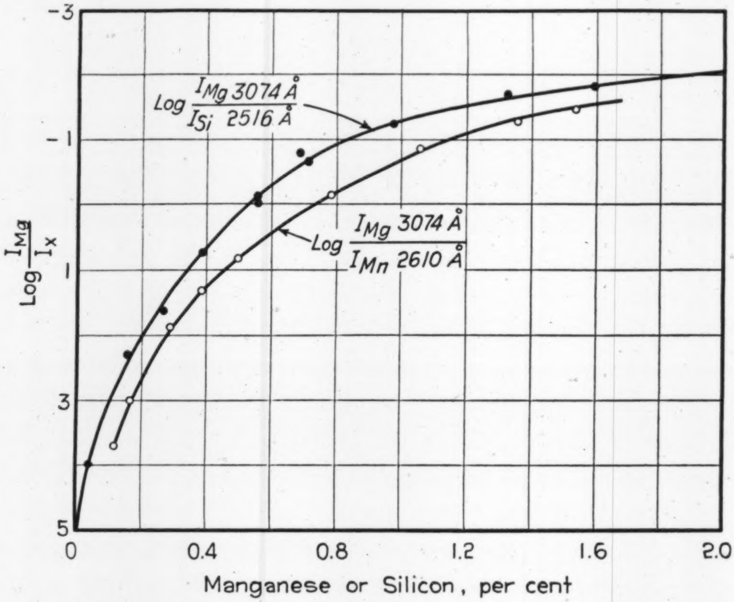

FIG. 1.—Representative Analytical Curves for Manganese and Silicon Analyses.

Analysis of an Alloy of Unknown Composition:

The analysis of an alloy of unknown composition is made by applying the logarithms of the relative intensities of the selected spectral-line pairs, determined in the manner described above, to the appropriate analytical curves and by reading therefrom the percentages of the elements under test present in the alloy.

Effect of Other Elements upon the Analysis:

It has frequently been found by different investigators(1,11) that the presence of other elements in the specimen markedly affects the spectrographic analysis for the element under test. However, no such effect has been found in the present analyses. The maximum concentrations of the other elements present in the Dowmetal alloys have no effect upon the analyses for manganese and silicon, thus making possible the use of the same analytical curves for the analyses of the various Dowmetal alloys.

ADVANTAGES OF SPECTROGRAPHIC ANALYSIS

One of the greatest advantages of this type of analysis is its rapidity. This is pronounced in the analysis of one specimen for several elements and especially so in the analyses of several specimens for one or more elements, as is usually the case in routine practice. This saving of time, especially in the case of several analyses to be made at one time, is reflected in the decreased cost of the individual analyses.

TABLE I.—REPRESENTATIVE MANGANESE AND SILICON ANALYSES.

Alloy	Plate	Spectrographic Analysis	Chemical Analysis
MANGANESE, PER CENT			
9849	102	0.060; 0.063; 0.065	0.056; 0.06
9849	99	0.056; 0.060; 0.063	
9807-10	95	0.161; 0.167; 0.163	0.17; 0.15
9807-10	98	0.161; 0.159; 0.159	
6627	58	0.203; 0.218; 0.222	0.21; 0.22
6627	61	0.230; 0.209; 0.225	
9850-3	111	0.527; 0.639; 0.607	0.54; 0.54
9850-3	113	0.491; 0.548	
5939	67	1.616; 1.548; 1.491	1.53; 1.55
5939	113	1.403; 1.437	
SILICON, PER CENT			
9799-1	72	0.057; 0.065; 0.069	0.036; 0.089; 0.059
9809-1	84	0.117; 0.126; 0.111	0.16; 0.16
9809-1	94	0.131; 0.125	
9795-12	91	0.188; 0.190; 0.213	0.19
9807-6	90	0.279; 0.266; 0.286	0.27; 0.23
9807-6	98	0.241; 0.263; 0.277	
9850-2	111	0.617; 0.629; 0.619	0.66; 0.76
9850-2	119	0.618; 0.611; 0.595	
9799-4	72	1.333; 1.638; 1.610	1.13[a]; 1.40[a]; 1.59; 1.60

[a] These two samples were possibly not completely dehydrated.

Another important advantage of this method is the fact that a complete qualitative and semi-quantitative analysis of the alloy for its metallic constituents may readily be made by inspection of the same plate which is used for the quantitative determination of one or more elements.

Other advantages are: (1) a permanent record of the analysis is kept by means of the filed plate; and (2) the fact that only an extremely small amount of the specimen is used up in the analysis, which is of advantage particularly in the case of a special sample of small amount.

PRECISION AND ACCURACY OF ANALYSIS

Table I indicates the representative precision and accuracy of the method of spectrographic analysis as carried out in routine practice.

Each spectrographic value was obtained from one spectrum. The values obtained from different photographic plates for different portions of the alloy are as shown. The analyses are given to three decimal places, not to denote an accuracy of this degree, but to enable the precision of the analyses to be seen. The values obtained by different routine chemical analyses are also shown.

SUMMARY

Routine spectrographic analyses of Dowmetal alloys containing approximately 90 per cent magnesium have been developed for manganese in the range from 0.009 per cent to 1.5 per cent and for silicon in the range from 0.005 per cent to 2.0 per cent to a degree of precision and of accuracy equal to that of routine chemical analysis.

The distinct advantages of this method are: (1) rapidity, especially in the analysis of one alloy for several elements and of several alloys for one or more elements, as is normally the case in practice; and (2) the fact that a complete qualitative and semi-quantitative analysis for the metallic constituents of the specimen may be made from the same plate that is used for the quantitative analysis.

A condensed spark discharge between solid alloy electrodes is used for the excitation of the spectrum. Maintenance of the spark discharge for a definite time interval before exposure has been found to increase greatly the accuracy of analysis. The method of analysis employs magnesium, the major constituent of the alloy, as an internal standard. The logarithms of the relative intensities of selected pairs of spectral lines of magnesium and of manganese, and of magnesium and of silicon, respectively, are obtained from the measured densities of these lines by means of a density-logarithm of intensity calibration pattern placed on each plate by the use of a Hansen step-diaphragm. Analytical curves are drawn relating the logarithms of the relative intensities of the selected spectral lines to the percentages of the elements under test for a series of alloys of known composition in which the concentrations of the elements under test vary over the ranges required for the analyses.

The spectrographic analysis of an alloy of unknown composition is carried out by the application of the experimentally determined logarithm of the relative intensity of the selected pair of spectral lines, one of magnesium and the other of the element under test, to the appropriate analytical curve.

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(2) Walther Gerlach and Eugen Schweitzer, "Die Chemische Emissions-Spektralanalyse, Grundlagen und Methoden," Leopold Voss, Leipzig (1930).

(3) G. Hansen, "Intensitatsmarken auf photographischen Platten," *Zeitschrift für Physik*, Vol. 29, p. 356 (1924); "Die Feinstruktur der Balmerlinien," *Annalen der Physik*, Vol. 78, p. 558 (1925).

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(6) C. W. W. Read and L. W. Johnson, "On the Technique of Spectral Intensity Measurement, with Special Reference to Choice of Photographic Plate," *Philosophical Magazine*, Series VII, Vol. 11, p. 1152 (1931).

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(9) K. B. Thomson and O. S. Duffendack, "Photographic Method of Measuring the Relative Intensities of Spectral Lines," *Journal*, Optical Soc. America, Vol. 23, p. 101 (1933).

(10) F. Twyman and A. A. Fitch, "The Quantitative Analysis of Steels by Spectrum Analysis," *Journal*, Iron and Steel Inst. (London), Vol. 122, No. II, p. 289 (1930).

(11) F. Twyman and C. S. Hitchen, "Estimation of Metals in Solution by Means of Their Spark Spectra," *Proceedings*, Royal Soc. (London), Vol. A 133, p. 72 (1931).

DISCUSSION

Mr. W. R. Brode.[1]—May I ask whether any attempt was made to compare unknown manganese or silicon lines against magnesium lines without the use of the impressed density pattern? Would the accuracy of such a method be equal to the authors' method?

Messrs. J. S. Owens[2] **and T. M. Hess.**[2]—We have made a comparison of the accuracies obtainable with and without the use of the intensity calibration pattern upon each plate. This comparison was made by means of the same blackening measurements of spectra obtained by means of our usual technique, which has proved its analytical reliability. A typical example of such a comparison is shown in the accompanying Figs. 1 and 2, which are analytical curves for the determination of manganese in magnesium alloys obtained from the differences of the blackenings of the indicated spectral lines of manganese and of magnesium and from the relative intensities of the same lines. The plotted values designated by circles at the same percentage of manganese were obtained from spectra taken with different exposure times on the same photographic plate; while those designated by crosses were obtained from spectra of the same alloys on a different plate. The spread of the plotted points indicates the accuracy obtainable. It will be seen that by the use of blackenings only, the deviations from the mean are much greater (except for that percentage at which the blackenings of the comparison pair of lines are equal) than those obtained by the use of relative intensities.

The results of our comparison indicate that only under certain definite and restricted conditions the method of analysis using the comparison of the blackenings of spectral lines may yield an accuracy approaching that of the method using the relative intensities of the same spectral lines, obtained by an intensity calibration pattern placed upon each plate. These conditions are: (1) the maintenance of constant exposure times for all spectra, both for the determination of the analytical curves and for the subsequent analyses of the unknown samples; (2) the maintenance of identical development and processing technique for each plate; and (3) the restriction of the blackenings of the spectral lines used to the straight-line portion of the characteristic curve of the photographic plate. The first and third of these conditions allow but a very limited concentration range of the element under analysis to be determined under the same spectroscopic

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[2] Research Investigator, X-ray and Spectroscopy Dept., The Dow Chemical Co., Midland, Mich.

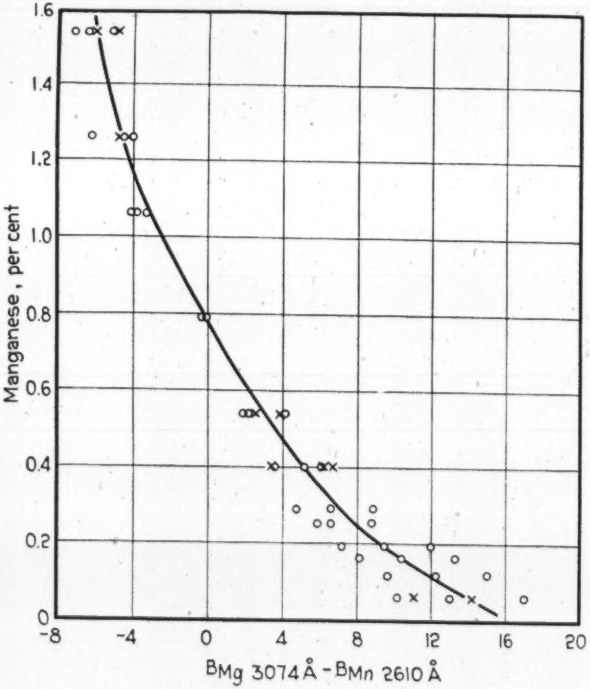

FIG. 1.—Analytical Curve for the Determination of Manganese in Magnesium Alloys by Use of the Blackenings of the Spectral Lines.

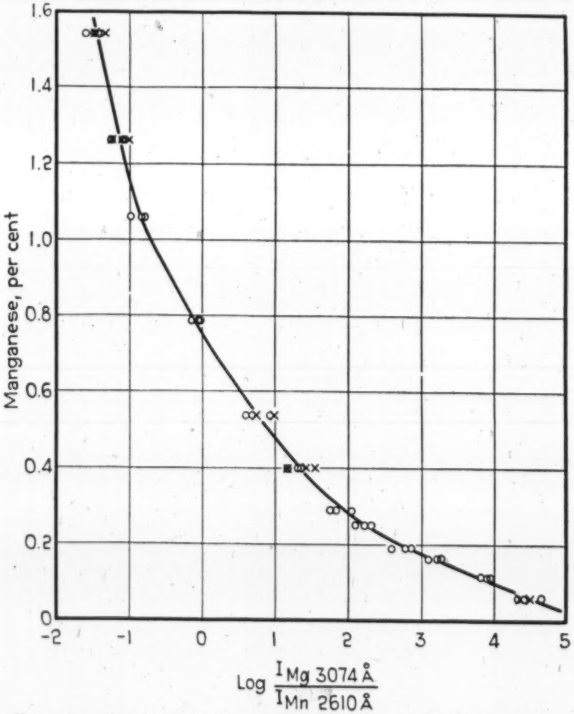

FIG. 2.—Analytical Curve for the Determination of Manganese in Magnesium Alloys by Use of the Relative Intensities of the Spectral Lines.

conditions. The first condition requires a preliminary knowledge of the percentage composition of the sample, to be obtained, for example, from a preliminary plate. The third condition is impossible of actual attainment because of the impossibility of determining the blackening limits of the straight-line portion of each plate without an intensity calibration thereon.

The fulfillment of the required conditions in analyses requiring the greatest accuracy necessitates a greater expenditure of time and effort than does the use of the relative intensities of the spectral lines. These conditions make the method based upon the comparison of the blackenings only, in our opinion, greatly inferior to the method based upon the relative intensities of the lines as a practical, accurate method of analysis.

MR. BRODE.—With the logarithmic sector, no photometer is necessary. There is an equal density at the extinction point on both lines. If the accuracy required is not greater than a certain value, it may be better to use a method of the sector type in which the measurement can be made in a very short time on a wet plate.

MR. OWENS.—I agree with Mr. Brode, provided the method used yields the necessary accuracy. However, our densitometer measurements can also be made on a wet plate. An economy of both material and time is also given by the fact that approximately three times as many spectra may be placed upon each plate by our method as by the logarithmic sector method. We must measure the densities of the spectral lines, but we simplify the photographing of the spectra by eliminating the use of the logarithmic sector.

SPECTROGRAPHIC DETERMINATION OF IMPURITIES IN COMMERCIAL CADMIUM

By Frances W. Lamb[1]

A brief survey of the production of metallic cadmium in the United States as published by the U. S. Bureau of Mines indicates the present status and growth of the cadmium industry. Starting with the year 1919, there were 100,000 lb. of cadmium produced in the United States. This amount has steadily increased, reaching a maximum in 1930 with a yearly production of 2,777,762 lb. There was a decline during the next few years; however, in 1933 the amount produced had returned to 2,276,933 lb., and in 1934 further increased to 2,777,384 lb., an increase of 22 per cent over the previous year and practically equal to that of 1930.

Cadmium is used principally in the electroplating, solder and paint industries. Of the total 1934 production, 1,800,000 lb. or 65 per cent was used in the electroplating of a variety of articles, from large objects such as caskets and burial vaults to the smallest pins. Of the cadmium electroplated products, about one-third are used in the automotive industries, the balance being scattered in practically all other lines. A comparatively new use of cadmium is in the manufacture of bearings from cadmium-base alloys.

The producers of cadmium have gradually increased the purity of their product from the former Silesian standard of 99.5 to 99.9 per cent, and in many cases, to 99.99 per cent. The common impurities found in commercial cadmium are: zinc, lead, iron, copper, thallium, nickel and tin. Obviously, the determination of impurities of this order by chemical methods in a routine control laboratory is difficult and somewhat uncertain. Moreover, the determination of small amounts of zinc in cadmium is a very long and tedious procedure and liable to error. A colorimeric method, which is relatively rapid, has been developed by Isbell[2] in which the violet precipitate formed upon the addition of a solution of mercuric ammonium thiocyanate to a neutral solution containing a small amount of zinc in the presence of a minute amount of copper is compared with a set of standards. A number of precipitation methods have been proposed, but in each case considerable difficulty is encountered in the attempt to make a complete quantitative separation of the relatively small amount of zinc from the large amount of cadmium. As a result, it is not uncommon to find the analysis reported by one laboratory to vary considerably from the analysis of the same material by another laboratory.

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[1] Metallurgical Dept., Bohn Aluminum and Brass Corp., Detroit, Mich.

[2] H. G. Isbell, "Rapid Determination of Zinc and Other Impurities in Cadmium," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 4, p. 284 (1932).

Since the percentages of impurities present in commercial cadmium are in the range most suitable for spectrographic determination, and since spectrographic analysis is not subject to the above-mentioned difficulties met with in chemical analysis, it has been the purpose of this investigation to determine the spectrographic procedure which would give the most uniform and satisfactory results for this particular problem and at the same time be simple in operation.

In the selection of a general procedure for spectrographic analysis, there are two principal features to be considered:

1. The method must permit rapid and uniform sampling.
2. The method must permit control of all factors influencing uniform volatilization of samples and standards.

The following general spectrographic procedures are in use:

1. Spark, using either metallic electrodes or solutions.
2. Arc, using either metallic electrodes, or graphite electrodes holding the sample either in the form of the metal, salt or solution.

A survey of the literature has revealed no publications on the spectrographic determination of impurities in cadmium. However, the problem is similar to that of the analysis of zinc, on which subject considerable spectrographic work has been done. Metallic electrodes have been used in the spectrographic analysis of zinc by Smith[1] who worked with both the arc and condensed spark procedures. Gerlach and Riedl[2] also used metallic electrodes in a study of the analysis of zinc by use of the interrupted arc, continuous arc, and condensed spark. While these authors have demonstrated that conditions for uniform exposures can be controlled when using metallic electrodes, yet these methods have certain disadvantages. During each exposure only a very small amount of the sample is volatilized and, consequently, there is danger of not obtaining representative sampling, especially if there is any segregation. Further, if metallic electrodes are used, either synthetic metallic standards must be prepared and then checked chemically or else standards may be selected from previously analyzed samples to cover the range desired. In either case, the standards are dependent upon accuracy of chemical analysis. The standards must then either be machined or cast in the form of electrodes and also each sample must be prepared in the same manner in order to obtain uniform results. The sampling is somewhat improved by the method used by Slavin[3] in which metallic zinc is dissolved in sulfuric acid, evaporated to dryness and the pulverized salt arced in graphite electrodes. However, most of the actual spectrographic analyses of zinc and zinc-base alloys have been accomplished by use of the solution method,[1,2,3] in which graphite electrodes are impregnated with an aliquot of a solution containing a definite concentration of the metal dissolved in acid. This procedure offers the best method for obtaining representative sampling and also the most suitable and convenient method for preparation of standards.

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[1] D. M. Smith, "The Spectrographic Determination of Cadmium, Lead and Iron in Zinc," *Transactions*, Faraday Soc., Vol. 26, p. 101 (1930).

[2] W. Gerlach and E. Riedl, "Spectro-analytical Examination of Commercial and Very Pure Metals," *Metallwirtschaft*, Vol. 12, p. 401 (1933).

[3] M. Slavin, "Analysis with Wedge Spectra," *Engineering and Mining Journal*, Vol. 134, p. 509 (1933).

EXPERIMENTAL

In using the solution method, the problem is that of developing a procedure for preparing and filling the electrodes that will insure uniform volatilization of the samples.

Since in the arcing of a sample any variation in volatilization produces variations in the intensity of the sensitive arc and spark lines, one of the most satisfactory criteria for judging whether or not several spectra represent uniform conditions of volatilization is to observe the intensity of a sensitive spark line of the base metal. A satisfactory cadmium line for this purpose is at wave length 2573.04 I.Å, which is very faint and any variation in intensity is readily detected by means of a low-power magnifying glass. While the above-mentioned line is too faint to be visible in the accompanying prints, yet the position is indicated. The cadmium line at wave length 2265.03 I.Å, is also a spark line; by comparing the relative intensity of this line with that of the adjacent arc line at wave length 2267.47 I.Å, further indication of the uniformity of volatilization is obtained; these lines are also indicated in the prints.

No doubt all who have worked with graphite electrodes impregnated with solutions have observed the abrupt change in the sound of the arc when the sample has been completely burned out. If the arc is permitted to burn a longer time the plate becomes slightly blackened by the light from the incandescent graphite electrodes. If the exposures are stopped when volatilization is complete, which is usually at the end of 40 sec. (for conditions which will be described under Procedure), the backgrounds of the spectra are very clear and the zinc lines at wave lengths 3345.0 and 3345.5 I.Å are faintly visible when only 0.001 per cent of zinc is present, while if the exposures are all timed at 60 sec., the backgrounds of the spectra are somewhat blackened and it is difficult to distinguish the above zinc lines in concentrations of less than 0.01 per cent. For this reason it is an important factor to discontinue the exposures as soon as complete volatilization has been accomplished. It is also desirable that this change in sound of the arc upon complete volatilization should be as marked and abrupt as possible. The length of time from starting of the arc to complete volatilization of the sample is also an indication of the uniformity of the conditions under which each sample is volatilized.

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[1] C. C. Nitchie, "Quantitative Analysis with the Spectrograph," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 1, p. 1 (1929).

[2] C. C. Nitchie and G. W. Standen, "An Improved Method of Quantitative Spectrographic Analysis," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 4, p. 182 (1932).

[3] M. L. Fuller and G. W. Standen, "Quantitative Chemical Analysis of Zinc and Zinc Alloys by Means of the Spectrograph," Procedure used by New Jersey Zinc Co.

Various shaped electrodes and methods for filling the electrodes have been studied for the purpose of determining, in accordance with the above criteria, which procedure gives the most uniform conditions of volatilization and which one gives the most decided change in sound of the arc upon complete volatilization. The electrodes used were $\frac{5}{16}$-in. Acheson graphite. For one set of experiments holes $\frac{1}{16}$ in. in diameter and $\frac{1}{8}$ in. deep were drilled in the graphite electrodes. With this small-size hole it was necessary to burn the electrodes in a 10-amp. arc for 1 min. to increase the porosity so that 0.1 ml. of solution would be readily absorbed by the electrode. This procedure has the advantage of burning out the impurities present in the graphite, which is necessary in order to determine less than 0.001 per cent iron. However, the point of complete volatilization was not abrupt but gradual and resulted in an undesirable blackening of the plate.

After trying other variations in procedure, the following method was found to give most satisfactory results and was, therefore, adopted. The electrodes are prepared by drilling a hole in one end with a standard $\frac{1}{4}$-in. 60-deg. center drill. These are then placed in an aluminum plate (similar to the one described by Petrey,[1] which is maintained at a temperature of 65 to 70 C. After the temperature of the electrodes has reached that of the plate, each one is filled with 0.1 ml. of solution. As soon as the liquid has completely disappeared the electrodes are transferred to a constant temperature oven set at 125 C., where they are left for 1 to $1\frac{1}{4}$ hr. By this procedure the intensity of the spark line at wave length 2573.04 I.Å, was observed to be very uniform for all exposures; also, the length of time for complete volatilization was very uniform, and the change in the sound of the arc upon complete volatilization was sufficiently abrupt.

PROCEDURE

Preparation of Standards.—A solution of pure cadmium is made by dissolving 125 g. in concentrated nitric acid and adjusting the volume to 500 ml. A solution of cadmium containing 0.50 per cent of each impurity may be prepared by dissolving 24.25 g. of pure cadmium plus 0.125 g. each of zinc, copper, lead, iron, thallium and nickel in concentrated nitric acid and adjusting the volume to 100 ml. The following standards were prepared from the above two solutions by a series of dilutions:

	ZINC, PER CENT	IRON, PER CENT	LEAD, PER CENT	COPPER, PER CENT	THALLIUM, PER CENT	NICKEL, PER CENT
Standard No. 1	0.001	0.001	0.001	0.001	0.001	0.001
Standard No. 2	0.005	0.005	0.005	0.005	0.005	0.005
Standard No. 3	0.010	0.010	0.010	0.010	0.010	0.010
Standard No. 4	0.025	0.025	0.025	0.025	0.025	0.025
Standard No. 5	0.050	0.050	0.050	0.050	0.050	0.050
Standard No. 6	0.100	0.100	0.100	0.100	0.100	0.100
Standard No. 7	0.250	0.250	0.250	0.250	0.250	0.250
Standard No. 8	0.500	0.500	0.500	0.500	0.500	0.500

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[1] A. W. Petrey, "Spectral Determination of Fluorine in Water," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 6, p. 343 (1934).

Preparation of Samples.—The samples are prepared in the same concentration as the standards, which corresponds to 0.25 g. of metal per 1 ml. If desired, a 10-g. sample may be used, in which case the sample is dissolved in concentrated nitric acid and the volume adjusted to 40 ml. For routine analysis the size of sample used is 2.50 g. in a volume of 10 ml.

Preparation of Electrodes.—The electrodes are prepared and filled as described above. The amount of solution added to each electrode is 0.1 ml., which corresponds to 25 mg. of the metal.

TABLE I.—SPECTRAL LINES USED.

Element	Wave Length in International Ångströms[a]	Range of Concentration, per cent	Neighboring Lines, Wave Length in International Ångströms
Zinc	3282.32	0.025 to 0.500	
	3302.6 3303.0	0.01 to 0.500	Sodium—3302.94[b]
	3345.0 3345.5	0.001 to 0.500	
Lead	2613.68 2614.204	0.001 to 0.500	Iron—2613.84[c]
	2802.007	0.001 to 0.500	
	2833.069	0.001 to 0.500	
	2873.32	0.005 to 0.500	
	4057.830	0.005 to 0.500	
Iron	2719.036	0.001 to 0.500	
	2720.909	0.005 to 0.500	
	2723.581	0.010 to 0.500	
	3020.645 3021.077	0.001 to 0.500	
Copper	2225.68	0.10 to 0.50	
	2227.76	0.10 to 0.50	
	2230.09	0.10 to 0.50	
	3247.548	0.001 to 0.500	
	3273.964	0.001 to 0.500	
Nickel	3101.561 3101.880	0.01 to 0.500	Iron 3099.901–3100.671[d]
	3414.77	0.001 to 0.500	
Thallium	2767.88	0.001 to 0.500	

[a] Wave length values taken from Tabelle der Hauptlinien Der Linienspektra Aller Elemente by H. Kayser.

[b] If sodium is present in the sample the doublet at wave lengths 3302.6 and 3303.0 I.Å, cannot be used; however, this interference is not likely.

[c] The iron line at 2613.84 I.Å interferes with the use of the lead lines 2613.68 and 2614.203 I.Å.

[d] The nickel lines at 3101.561 and 3101.880 I.Å, must not be confused with the iron lines at 3099.901 — 3100.671 I.A.

Conditions for Making Exposures.—The instrument used is the Bausch & Lomb medium quartz spectrograph equipped with complete illuminating unit, the sector disk of which is adjusted to allow $\frac{1}{32}$ of the light to reach the slit. The arc is operated at 11 amp. on a 220-v. direct current line. The lower electrode, containing the sample, is made the positive electrode. The exposures are discontinued as soon as the samples are completely volatilized, the time usually required is 40 sec. The images of the two electrodes formed by the quartz spherical condensing lens are kept at a fixed distance apart, one above and one below the slit.

Evaluation of Plates.—The "comparison sample" method is used for estimating the percentage of impurities occurring in the samples. For this purpose, the spectra are arranged on the plate as shown in Fig. 1 (*b*), so that the intensities of the lines in the spectra of the samples can be compared directly with the intensities of the lines in the spectra of the standards. By this method the percentage of each impurity in the sample can be determined with certainty between limits, and can be estimated to a fair degree of accuracy within limits. The arc spectrum of cadmium does not have lines suitable for use in making evaluations by the "internal standard" method. While the "comparison sample" method involves more work than the "internal standard" method for the analysis of each sample, yet the former method has proved to be very satisfactory for this problem. Spectral lines which gave suitable gradation in intensity with variations in concentration of the respective impurities under the above conditions were selected for use in making determinations by the "comparison sample" method. These lines are indicated on the standard plate shown in Fig. 1 (*a*). Considerable detail is lost in the process of reproduction; however, the gradation in intensity of the lines visible in the print for the higher concentration of impurities is likewise visible in the original plate for the lower concentrations. The spectral lines used are listed for each impurity in Table I.

In addition to the elements listed, the spectra are always examined for other impurities and in particular for tin, a trace of which is injurious to cadmium-base bearings and causes streakiness in cadmium plating.

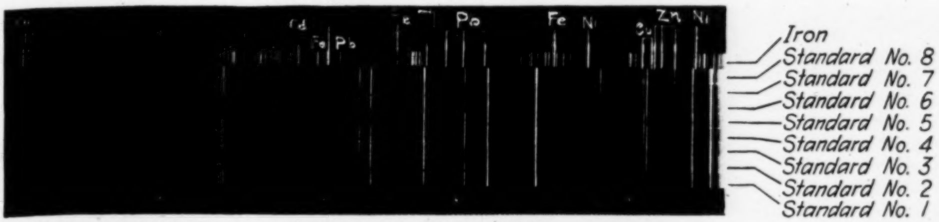
Figure 1 (*a*) is a spectrogram of the cadmium standards used for the estimation of impurities in commercial cadmium.

Figure 1 (*b*) is a reproduction showing the arrangement of the spectra of standards and samples for making an analysis. Sample No. 1 is a very impure commercial cadmium, the analysis of which is given below.

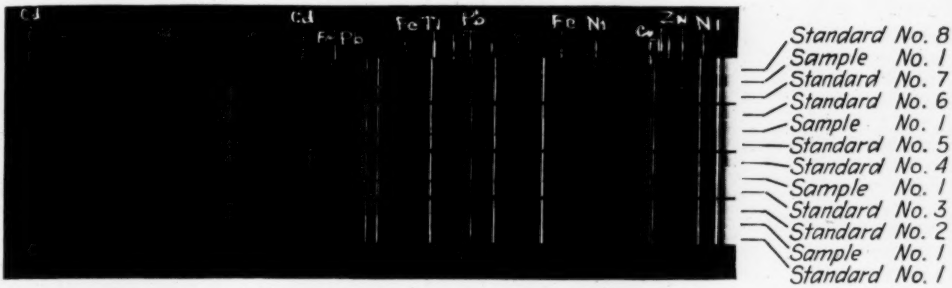
The three spectra in Fig. 1 (*c*) represent three grades of commercial cadmium, the spectrographic analyses of which are:

	ZINC, PER CENT	IRON, PER CENT	COPPER, PER CENT	LEAD, PER CENT	NICKEL, PER CENT	THALLIUM, PER CENT	TIN, PER CENT
Sample No. 1....	0.35 to 0.40	less than 0.005	0.030	0.20	0.025	0.001	trace
Sample No. 2....	0.003	0.005	0.001	0.02	less than 0.001	0.040	.....
Sample No. 3....	0.001	less than 0.005	0.025	0.01	less than 0.001	less than 0.001	.....

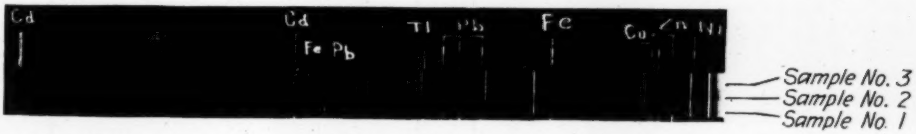
The presence of thallium in cadmium to be used for electroplating is to be avoided since an amount as small as 0.001 per cent causes blistering. Spectrographic analysis offers a ready control method for checking the percentage of thallium. Of the large number of samples analyzed, sample No. 2 was found to have the largest amount of thallium.


Cd
Fe Pb
Fe Ti
Pb
Fe Ni
Co
Zn Ni
Iron
Standard No. 8
Standard No. 7
Standard No. 6
Standard No. 5
Standard No. 4
Standard No. 3
Standard No. 2
Standard No. 1

(*a*) Spectra of cadmium standards for analysis of commercial cadmium.

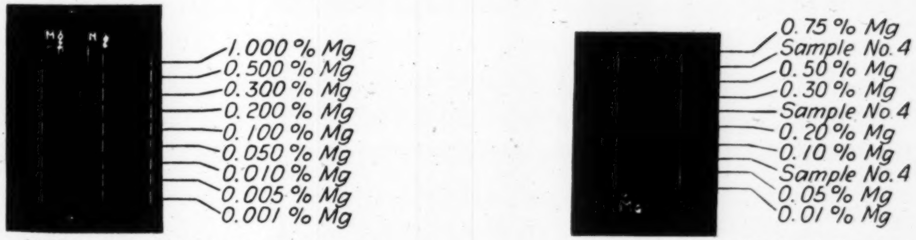

Cd
Cd
Fe Pb
Fe Ti
Pb
Fe Ni
Co
Zn Ni
Standard No. 8
Sample No. 1
Standard No. 7
Standard No. 6
Sample No. 1
Standard No. 5
Standard No. 4
Sample No. 1
Standard No. 3
Standard No. 2
Sample No. 1
Standard No. 1

(*b*) Spectra showing typical analysis of sample of commercial cadmium.


Cd
Cd
Fe Pb
Tl
Pb
Fe
Co
Zn Ni
Sample No. 3
Sample No. 2
Sample No. 1

(*c*) Spectra of three grades of commercial cadmium.

FIG. 1.—Spectrograms of Cadmium.


Mg
Ni
1.000 % Mg
0.500 % Mg
0.300 % Mg
0.200 % Mg
0.100 % Mg
0.050 % Mg
0.010 % Mg
0.005 % Mg
0.001 % Mg
0.75 % Mg
Sample No. 4
0.50 % Mg
0.30 % Mg
Sample No. 4
0.20 % Mg
0.10 % Mg
Sample No. 4
0.05 % Mg
0.01 % Mg

(*a*) Spectra of cadmium standards for analysis of magnesium.

(*b*) Typical analysis of cadmium-base bearing alloy for magnesium.

FIG. 2.—Spectrograms of Cadmium for Analysis of Magnesium.

OTHER APPLICATIONS

Cadmium Alloys.—This same general procedure may be used for cadmium alloys. The method has been found very suitable for the rapid control analysis of magnesium in one of the cadmium-base bearing alloys. Figure 2 (*a*) shows the spectra of cadmium standards for determining magnesium content, and Fig. 2 (*b*) presents a typical analysis of a cadmium-base bearing alloy, which was found to have 0.15 per cent magnesium by spectrographic analysis. In addition to being more rapid than the chemical method for determining magnesium, the spectrographic method has the advantage of detecting all impurities which may be present at the same time.

The spectral lines examined for the determination of magnesium in cadmium-base alloys are tabulated below:

WAVE LENGTH IN INTERNATIONAL ÅNGSTRÖMS	RANGE OF CONCENTRATION, PER CENT
2776.71	0.10 to 1.00
2778.29	0.10 to 1.00
2779.85	0.01 to 0.50
2781.43	0.10 to 1.00
2782.988	0.10 to 1.00
2795.540	0.001 to 0.10
2802.712	0.001 to 0.10

Cadmium Compounds.—A further application of this same general procedure is in the analysis of cadmium compounds. For example, an amount of cadmium oxide corresponding to 2.50 g. of cadmium is dissolved in concentrated nitric acid, the volume made up to 10 ml. and the analysis made in the same manner as with solutions of metallic samples.

SUMMARY

A spectrographic procedure for analysis of the impurities in commercial cadmium is described and illustrated by reproductions of typical analyses.

A description of a simple method developed for preparing and impregnating graphite electrodes with solutions, which insures the obtaining of uniform exposures with minimum blackening of the background of the spectra, is given.

The application of this procedure to the analysis of cadmium-base alloys and to cadmium compounds is pointed out.

Acknowledgment.—The author wishes to express her appreciation for the cooperation received from Dr. E. J. Martin of General Motors Research Laboratories, and from Mr. L. K. Lindahl of the Udylite Co.

THE PURIFICATION OF GRAPHITE ELECTRODES FOR SPECTROCHEMICAL ANALYSIS

By G. W. STANDEN[1] and L. KOVACH, JR.[2]

Graphite rods are commonly used as arc electrodes in spectrochemical analysis. The reliability of a spectrographic analysis depends largely on the purity of the electrodes used. It is necessary, therefore, to use the purest form of carbon obtainable.

Regraphitized graphite rods for spectrographic use are obtainable. These electrodes are far superior to the ordinary grade of graphite, and are sufficiently pure for much spectrographic work. Occasional traces of aluminum, boron, calcium, copper, iron, magnesium, manganese, silicon, titanium, and vanadium normally are present. These impurities sometimes may be the source of error in a spectrochemical analysis. This is particularly true for the present-day high-purity commercial metals, such as zinc.

The qualitative detection of impurities in a sample will, of course, be uncertain with respect to those elements present in the electrodes. In making a quantitative determination for any element that is present as an impurity in the electrodes, the final result will be in error to the extent of the amount present in the electrodes. Accordingly it has become necessary to obtain a purer electrode for spectrochemical analysis.

In order to eliminate such difficulties two methods of effecting the necessary purification have been devised and successfully used in this laboratory whereby all the elements previously mentioned, with the exception of boron and silicon, are removed. One method relies principally on heating the electrodes in atmospheres of ammonia and chlorine successively, and the other method employs various solvents for leaching out the impurities. Using either method, a final treatment is given by burning in an electric arc.

Heat-Treatment Method:

Electrodes which have been given a brief preliminary solvent treatment are maintained in a furnace at 1000 C. for 23 hr. During this period the electrodes are initially saturated with dry ammonia and finally maintained in an atmosphere of chlorine for the remainder of the run.

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[1] Spectroscopic Investigator, Research Division, The New Jersey Zinc Co., Palmerton, Pa.

[2] Laboratory Assistant, X-Ray and Spectrographic Laboratory, Research Division, The New Jersey Zinc Co., Palmerton, Pa.

The furnace used in this method is shown in Fig. 1. The furnace consists of a vertical silica tube 25 in. long, $5\frac{3}{4}$ in. in inside diameter, and $6\frac{1}{4}$ in. in outside diameter, closed at the bottom. The furnace tube is wound with 160 ft. of No. 12 gage (0.081 in. in diameter) nichrome wire for a distance of about 20 in. upward from the base of the tube. The cover for this tube is made in two pieces. The lower or plug part is made of graphite $5\frac{1}{2}$ in. in diameter and 8 in. long. The upper or flange part is made of carborundum 8 in. in diameter and 1 in. thick. The flange is bolted to the plug by means of two graphite bolts $\frac{3}{4}$ in. in diameter by 3 in. long. The bolts screw into threaded holes in the graphite plug. The heads of the bolts are protected from disintegration by a heavy coating of refractory cement. If the cover is made entirely of graphite that portion that is exposed to the air disintegrates. Three holes are drilled vertically through the cover to accommodate a silica entrance tube for the gases, a silica pyrometer tube, and a silica exhaust tube. The temperature of the furnace is maintained between 1000 and 1100 C.

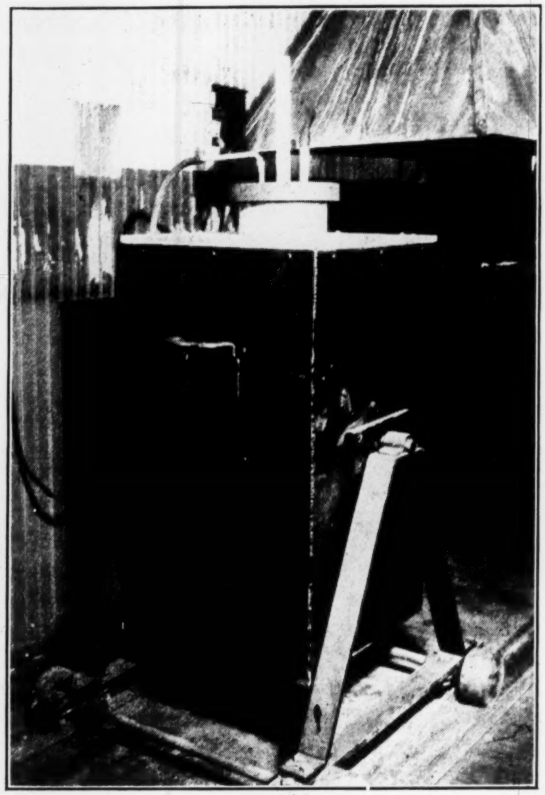

FIG. 1.—Electrode Purification Furnace.

The procedure is as follows: The graphite rods[1] are cut into the size and form in which they are to be used as electrodes in the analytical procedure. The electrodes are placed in a Pyrex beaker and covered with an equal volume mixture of concentrated nitric and hydrochloric acids. They are boiled in this solution for about 2 hr. and then washed twice with distilled water. They are then covered with concentrated ammonium hydroxide, the beaker placed on a warm plate and the electrodes allowed to soak in this solution for 2 or 3 hr. at a temperature of about 50 C. The ammonium hydroxide is then poured off and the electrodes are dried in the beaker on a hot plate.

The furnace is filled about one-third full with these electrodes. In using 2-in. lengths of $\frac{5}{16}$-in. diameter graphite rod, approximately 600 electrodes can be run in each batch. By filling the furnace no more than one-third full and by using the deep graphite cover described above, it is found that oxidation of the electrodes is minimized and the purification is more uniformly effected throughout each batch of electrodes.

As soon as the electrodes have been placed in the furnace the graphite cover is put on and the silica tube for the gases, the pyrometer tube, and the exhaust tube are put into position. In order to insert the gas tube, which extends to the bottom of the furnace, the furnace is tilted on its side allowing the electrodes to drop away from the wall so that the tube can be placed in position. The gas tube is connected by means of rubber tubing to the ammonia tank and this gas is passed into the furnace at the rate of about 3000 to 4000 cc. per min. for about 15 min. or until the electrodes are saturated. This is determined by testing the gases passing from the exhaust tube for ammonia. The ammonia is then shut off, the gas tube connected by means of another rubber tube to the tank of chlorine and the latter is passed in at the rate of 150 to 200 cc. per min. for the remainder of the run. About 23 hr. is allowed for each run, after which time the chlorine is shut off, the tubes and cover removed and the electrodes dumped into a clean graphite container to cool.

The time required for this heat treatment is less for a smaller size furnace. In preliminary laboratory experiments with a 1-in. diameter tube furnace, only 1 hr. was required.

Solvent-Purification Method:

After they have been cut into the size and form desired for use, the graphite electrodes are subjected to a series of solvent treatments which effectively leach out the various impurities with the exception of boron and silicon.

The electrodes are placed in a Pyrex beaker and covered with an equal volume mixture of concentrated nitric and hydrochloric acids.

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[1] The rods used in the work described in this paper were made of a regraphitized graphite especially prepared for spectrographic use. They were obtained from the Acheson Graphite Co., Niagara Falls, N. Y.

The solution is boiled for 2 or 3 hr., the beaker is placed on a warm plate and the electrodes allowed to soak for at least 20 hr. at a temperature of about 70 C. The acid is then poured off, the electrodes washed with distilled water and covered with concentrated ammonium hydroxide. The solution is heated to boiling and the electrodes are allowed to remain in this solution for about 20 hr. at a temperature of about 50 C. on a warm plate. The electrodes are washed with distilled water and covered with glacial acetic acid. This solution is heated to boiling, the beaker moved to a warm plate and the electrodes soaked for 20 hr. at a temperature of about 70 C. They are then boiled for 1 hr. in distilled water. Finally the electrodes are again treated with hot concentrated ammonium hydroxide for 30 min., washed with distilled water, and dried.

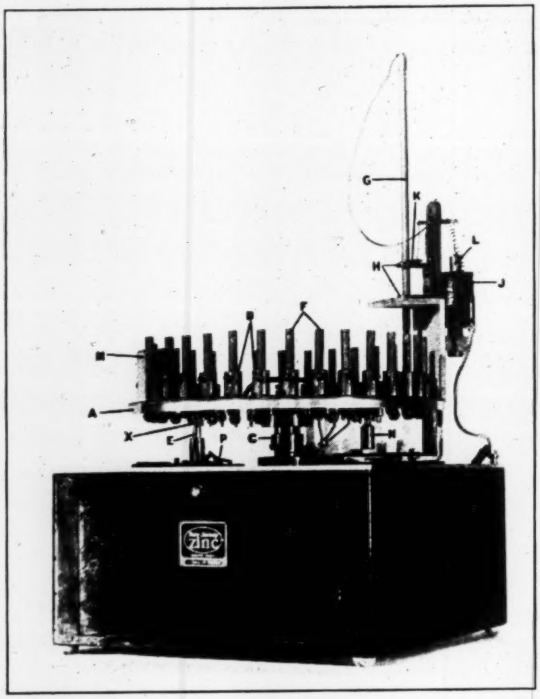

FIG. 2.—Automatic Electrode Burning Machine.

The foregoing procedure may seem at first glance to be unnecessarily complicated. This method was developed by trial, however, and subsequent investigation showed that the omission of one or more of the steps of the procedure or the departure from the order of treatments used lessened its effectiveness.

Electric-Arc Treatment:

It has been our experience that the highest purity is obtained when the electrodes are burned in an electric arc following either of the purification procedures given above. By this treatment traces of remaining impurities or impurities with which the electrodes may have become contaminated in handling are volatilized.

This treatment consists merely of burning each of the previously purified electrodes in an electric arc for about $1\frac{3}{4}$ min. using a current of 10 to 15 amp. The treated electrodes are used in the positive electrode position in the circuit.

In laboratories where a large number of electrodes are constantly needed, this final burning treatment would necessarily require considerable manual labor. In order to eliminate the constant attention normally required by this step in the procedure an automatic electrode burner has been devised by this laboratory. This device burns successively 31 electrodes under the required conditions without the attention of an operator. At the end of the run the device automatically stops and the burned electrodes must be replaced with a new set to be treated.

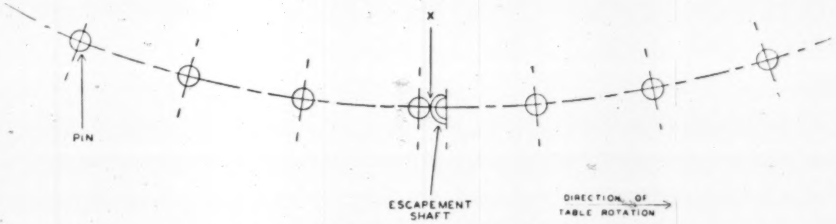

FIG. 3.—The Escapement Mechanism.

This instrument, shown in Fig. 2, consists of a revolving table, *A*, 1 ft. in diameter and made of $\frac{3}{8}$-in. duralumin with a $\frac{1}{8}$-in. transite top covering. Around the circumference of the table, $\frac{3}{4}$ in. from the outer edge, are bolted 31 electrode holders, *B*, made of brass. These holders are spaced 1 in. apart from center to center. This table is connected to a central shaft *C* of steel 1 in. in diameter which extends down into the box into a steel housing bolted to the $\frac{3}{16}$-in. thick duralumin cover of the box. The lower end of the shaft rests on a ball bearing.

On the underside of the revolving table is mounted a circle of steel pins, *D*, one pin on the same radial line with each electrode holder. A shaft, *E*, with a special axial slot projects vertically through the top of the metal cover to a height sufficient to engage the pins beneath the rotating table. A cross-section of this shaft and of one of the pins is shown in Fig. 3. The shaft is driven through fiber gears by a synchronous clock motor at the rate of one revolution in $1\frac{3}{4}$ min. The combination of slotted shaft and pins constitutes an escapement mechanism whereby the table may be allowed to rotate intermittently the distance between adjacent electrodes. The operation of this mechanism is as follows: At the start of a cycle one of the pins is held against the slotted shaft at the point *X*, Figs. 2 and 3. A spiral spring attached to the central supporting shaft of the table supplies the tension to hold the pin against the slotted shaft. The slotted shaft rotates 180 deg. until the pin falls into the slot. On a second rotation of 180 deg. the pin is released, allowing the next pin to come up to position *X* as mentioned above, etc. During the period of $1\frac{3}{4}$ min. while an individual pin is in contact with the slotted shaft, an electric arc is burning between one of the positive electrodes, *F*, on the table and a stationary negative electrode, *G*, mounted above it. The intermittent rotation of the table successively brings fresh electrodes under the stationary one.

The upper graphite electrode is held in a vertical position by a steel guide *H*. This electrode, however, is movable vertically and the movement is controlled through the use of a solenoid *J* and the grip *K*. In starting, the upper and lower electrodes are in contact. When the current is passed through the circuit the solenoid is energized, drawing the armature *L* downward which in turn pulls the upper electrode up to a distance of about $\frac{1}{4}$ in., thereby starting an arc between the electrodes. At the end of the arcing period the table moves, drawing the lower electrode away from the upper electrode. By the time the next lower electrode has come into position underneath the upper electrode the gap between the arcing electrodes is too large to maintain the arc. When the arc breaks, the current is simultaneously cut off from the solenoid, permitting the upper electrode to fall into contact with the lower electrode. The contacting of the electrodes completes the circuit and the arcing cycle is repeated. In the space between the first and thirty-first electrode holder is mounted, on the table, a transite block, *M*, 2 in. high by 1 in. square. After the last electrode has been burned the upper electrode falls on this transite block instead of on another electrode.

In order to prevent the cycle of burning operations from being repeated, an automatic switch is used to stop the escapement mechanism and to permanently break the arc circuit. As the table moves to this final position the post *N* mounted on the underside of the table depresses a trip lever *P* which springs open a switch inside the box. This is a four-pole switch connected in both the 110 a.c. clock motor circuit and in the 220 d.c. arc circuit. After the automatic switch is opened, further rotation of the table is blocked by the interception of the pin by the escapement shaft. Since the latter shaft has ceased to rotate, further motion of the table is prevented.

Before starting the burning of the next set of electrodes the spiral spring connected to the supporting shaft of the table must be wound. This shaft has a slotted coupling integral with it so that the table can be lifted free of the escapement mechanism for this winding, which is accomplished by rotating the table top one revolution.

Relative Merits of the Two Methods:

From the standpoint of the efficacy of the purification the two methods are equally satisfactory. The choice of method will depend largely upon the personal preference of the user and the facilities at hand. From the experience of this laboratory the heat-treatment method requires less time and is less expensive than the solvent method.

Acknowledgment.—The authors wish to express appreciation for assistance obtained from other members of the Research Division, The New Jersey Zinc Co., during the course of the investigation and in the preparation of the manuscript.

DISCUSSION

Mr. Eugene Wainer.[1]—How much time does the entire procedure consume, and what is the volume or number of electrodes that may be treated in a single operation?

Mr. M. L. Fuller.[2]—Our routine practice is to spend several weeks periodically purifying a large quantity of electrodes. The heat treatment process requires one day to treat 600 electrodes. Thirty electrodes an hour can be treated with the automatic electrode burner.

Mr. Wainer.—How long does it take to remove vanadium by the electrode treatment?

Mr. Fuller.—We designed the procedure so that it removes all the impurities. We do not know whether one is volatilized more rapidly than the other and accordingly have no information on the length of time necessary to remove vanadium. Vanadium must come off more readily than some other impurities, since it has been observed that whenever a batch of heat-treated electrodes had not been rendered free of impurities, vanadium was never one of those present. Copper, iron and magnesium are the elements most difficult to remove.

Mr. Wainer.—Vanadium is a most persistent impurity in the usual spectrographic carbon. Since vanadium occurs in practically all minerals and consequently in most finished products, a pure and vanadium-free electrode such as Mr. Fuller describes is of considerable value for this particular element. In the past we have attempted to utilize, for vanadium analysis, electrodes prepared from sugar carbon, but their lack of mechanical strength is a great disadvantage.

Mr. W. R. Brode.[3]—Were the original carbons the regular graphite carbons?

Mr. Fuller.—The original carbons used are the regular grade of Acheson spectroscopic graphite and not the more expensive grade.

Mr. Brode.—Would the cost of purification run high, so that it would be more economical to buy the other type?

Mr. Fuller.—The purification of Acheson electrodes a little more than doubles our electrode cost, but this total cost is much less than the cost of electrodes of equivalent purity which are on the market.

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[1] Spectroscopist, Titanium Alloy Manufacturing Co., Niagara Falls, N. Y.

[2] Investigator, Research Division, The New Jersey Zinc Co., Palmerton, Pa.

[3] Associate Professor of Chemistry, Ohio State University, Columbus, Ohio.

QUANTITATIVE ANALYSIS OF NON-FERROUS ALLOYS BY SPECTROSCOPIC METHODS

By R. A. Wolfe[1]

Quantitative chemical analyses for certain substances are often difficult to make with accuracy and rapidity. The advantage of using the spectrograph for determining some of these substances is fast becoming recognized. Various methods have been devised since Lockyer[2] first estimated the abundance of an element in the electrodes of an arc by observing the strength of certain spectral lines. Until recently, however, all of the methods for quantitative spectrographic analysis were merely rough estimates. Even rough estimates were often of great value for research experiments and for aiding the chemist in determining the probable range of analysis to expect. It is not hard to understand how the accurate methods of spectrographic analysis, the commercial use of which is just beginning, can be of very great value in the development of new products and in the improvement of old ones because of the ease and quickness with which such analyses can be made.

The method of spectrographic analysis devised by Duffendack, Wolfe and Smith[3] is the most accurate and one of the quickest yet discovered. It has been used in obtaining the data discussed in this paper. In order to use this method successfully three steps are necessary:

1. Fixing of excitation conditions.
2. Determination of a working curve.
3. Analysis of the unknown specimen.

In fixing the excitation conditions, the arc or spark must first be standardized. It has been found that after a certain current through an arc is exceeded, additional arc current shows very little change in the relative intensities of a selected pair of spectral lines. The curve in Fig. 1 shows the relation between arc current and relative intensity of a pair of lines in the spectrum of a nickel-chromium alloy. The lines used were chromium 4289.73 Å and nickel 4401.55 Å. For this particular alloy arc currents of 10 amp. or above would be satisfactory. Although an arc source has been used for these investigations a spark source may be used and standardized by the method of Gerlach and Schweitzer.[4]

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[1] Research Physicist, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

[2] W. Lockyer, *Transactions*, Royal Soc. (London), Vol. 164, II, p. 479 (1874).

[3] O. S. Duffendack, R. A. Wolfe and R. W. Smith, "Quantitative Analysis by Spectroscopic Methods," *Industrial and Engineering Chemistry*, Vol. 5, July 15, 1932.

[4] Gerlach and Schweitzer, "Chemische Emissions Spektralanalyse, Grundlagen and Methoden," Leopold Voss, Leipzig (1930).

For determining the working curve, a series of alloys, solutions, or mixtures is made in which the percentage composition of the material to be determined is varied over the range chosen for the analysis. Using the proper excitation conditions, spectrograms are then made of the series of known alloys, solutions or mixtures, together with an intensity pattern for each photographic plate used. For the intensity pattern, the calibrated step diaphragm is used and may either be one of the Hansen[1] type or better one of the type devised by Thomson and Duffendack.[2] The value of the logarithm of the relative intensities of the selected lines is determined for each alloy in the series with the intensity patterns serving to calibrate the plates. The ratio of the logarithms of the relative intensities of such spectral lines is then plotted against the known composition to form the working curve. It should be emphasized that after a working curve has once been determined for any given type of analysis it is unnecessary to refer again to known standards.

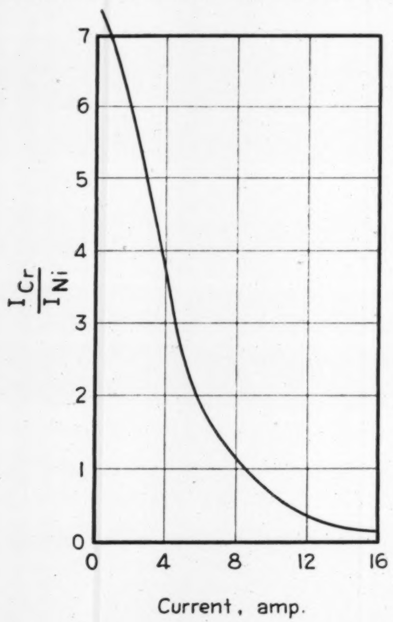

FIG. 1.—Relation Between Arc Current and Relative Intensity of a Pair of Lines in Spectrum of a Chromium-Nickel Alloy.

The analysis of an unknown specimen is simple. The spectrum of the unknown sample is photographed in the manner described and the logarithm of the relative intensities of the selected lines is determined. The logarithm of the relative intensities of the spectral lines is applied to the working curve and the percentage read on the scale.

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[1] G. Hansen, *Physik*, Vol. 29, p. 356 (1924).

[2] K. T. Thomson and O. S. Duffendack, *Journal*, Optical Soc. America, Vol. 23, p. 101 (1933).

Determination of Aluminum in Nickel Alloys:

The excitation conditions were first determined for an arc source and a curve similar to the one shown in Fig. 1 resulted. Values of arc current greater than about 10 amp. were found to be satisfactory. Actually an arc current of 15 amp. was used so that a greater quantity of the unknown material could be consumed. The electrodes were about ¼ in. in diameter. Next a working curve was determined. In order to do this, the sensitivity of several aluminum lines to changes in the abundance of aluminum was studied and the line at 2660 Å was selected for the analysis. This was compared in intensity with the nickel line at 2746 Å. The ratio of the intensity of these two spectral lines was measured in the spectra of a number of aluminum alloys which had been analyzed chemically and the logarithm of the ratio was plotted against the abundance of aluminum as described in the method given above. The curve is shown in Fig. 2.

While taking the data for the curve shown in Fig. 2, a difficulty was met which is quite common in studies of this kind. The spectrograph used was of the Littrow type and with this type of mounting light is often reflected from the front surface of the lens, and when this amount is excessive, this light will fog the photographic plate. Intensity measurements on plates having background cannot be made accurately and some means of eliminating this defect must be used. This can often be accomplished by changing the setting of the prism slightly, placing suitable diaphragms inside the spectrograph case, or tipping the lens. Clear spectrograms are necessary for measuring the relative intensities of spectral lines accurately.

The curve shown in Fig. 2 was developed to analyze for aluminum in amounts from 1 to 5 per cent. If analyses for smaller amounts of aluminum are demanded, an aluminum line of greater sensitivity would be necessary.

Determination of Magnesium in Zinc Alloys:

Because of the much lower melting point of zinc alloys, large arc currents are difficult to use. It was found that a stable arc could be maintained on an arc current of 4 or 5 amp. without undue melting of the electrodes. The electrodes used were about ½ in. in diameter and a large quantity of the material was consumed during an exposure. Exposure times were approximately 30 sec. The analytical range desired was from 0.005 to 0.05 per cent magnesium. A careful study of the lines available showed the magnesium line at 3832.17 Å to be suitable. This line shows ample sensitivity in the analytical range desired. The zinc line at 3058.8 Å was used as a standard for comparison.

The working curve is shown in Fig. 3. While the percentage range for magnesium is shown only as high as 0.05 per cent, the curve could undoubtedly be extended to considerably higher values. For determining very small concentrations of magnesium, the magnesium line at 2852.13 Å was

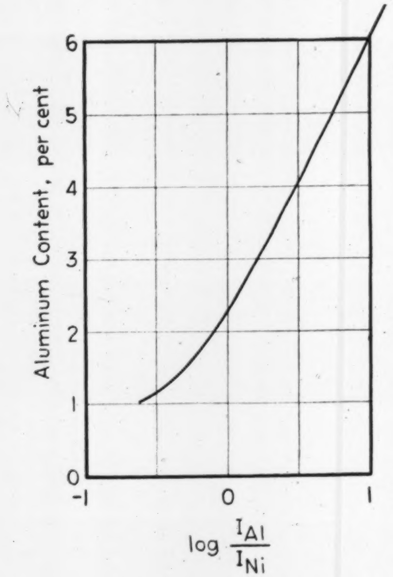

FIG. 2.—Relation Between Percentage of Aluminum in Aluminum-Nickel Alloys and Logarithm of Relative Intensity of a Pair of Lines in the Spectra of the Alloys.

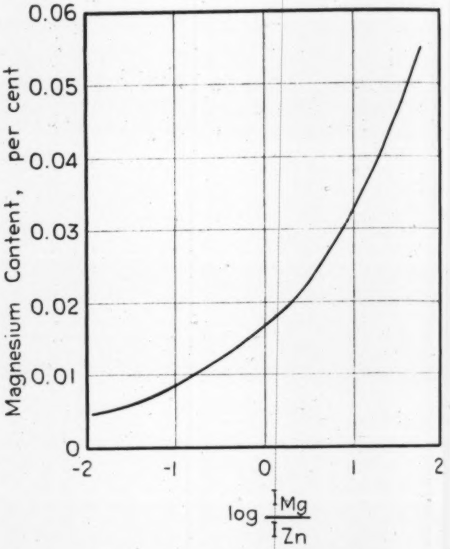

FIG. 3.—Relation Between Percentage of Magnesium in Magnesium-Zinc Alloys and Logarithm of Relative Intensity of a Pair of Lines in the Spectra of the Alloys.

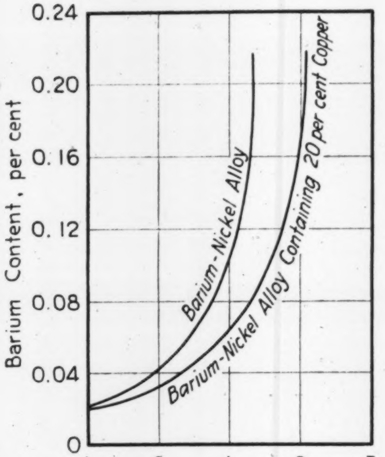

FIG. 4.—Relation Between Percentage of Barium in Barium-Nickel Alloys and Logarithm of Relative Intensity of a Pair of Lines in the Spectra of the Alloys.

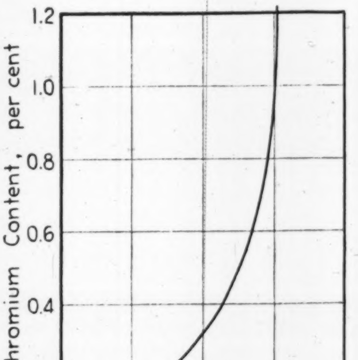

FIG. 5.—Relation Between the Percentage of Chromium in Chromium-Nickel Alloys and Logarithm of Relative Intensity of a Pair of Lines in the Spectra of the Alloys.

used and compared in intensity with the zinc line at 3035.8 Å. This line is much too sensitive for large amounts of magnesium and should not be used for concentrations much above 0.01 per cent. No difficulty was experienced in check analyses or in experimental technique.

Determination of Barium in Nickel Alloys:

The analysis of barium in barium-nickel alloys has been carried out over a period of several years. Enough checks have been made against chemical analyses to show that the spectrographic method of analysis is as reliable as the chemical method. Two working curves are shown in Fig. 4. The working curve for barium-nickel alloys containing 20 per cent copper was included to illustrate a condition frequently encountered in determinations of this kind. In both of these curves, the same pair of barium and nickel lines was used, and it is obvious that the addition of copper to the alloy affects the variation of the relative intensities of these lines with the percentage of barium. Some lines of an element are very strongly affected by the presence of small amounts of other elements in the same alloy, while other lines of the same element are not. Sometimes the presence of certain elements in the alloy may affect the entire spectrum of the element being analyzed. This may be due to a change in the abundance of the element in the arc due to an increase in vapor pressure of the element added to the alloy. This is especially true for those elements easily placed in a vapor condition. Still another cause of changing line intensity is possible in those cases where close resonance exists between excited states of two elements, and thus it is necessary, when a mixture of elements is used, to examine the lines chosen for such effects. Too little is known as yet about the influence of additional elements on the accuracy of spectrographic analysis, and much work is desirable in this field. With the knowledge available at present, it is necessary to make separate working curves for each alloy analyzed.

Determination of Chromium in Nickel:

The excitation conditions were fixed as usual, the curve of Fig. 1 showing the correct arc current to use. A large number of satisfactory chromium lines are available, the one at 4289.73 Å being chosen only because its sensitivity was good in the analytical range desired. The nickel comparison line was 4401.55 Å.

On account of the change in contrast of the photographic plate with wave length, it is best to select lines for analysis reasonably close together. If such lines are widely separated, a calibration pattern for each line used is necessary (Thomson and Duffendack[1]). The working curve for chromium in chromium-nickel is shown in Fig. 5. This curve is of the same general form as the others shown and is the type of curve to be expected. An exception to this type of curve may occur when the unknown alloy contains large quantities of a substance easily vaporized.

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[1] *Loc. cit.*

It should be pointed out that the method of spectrographic analysis outlined above is satisfactory only when certain conditions are rigidly adhered to. The procedure is built upon a correct method of photographic photometry. The calibration of each plate is a necessary and essential feature of the method. Because of the calibration pattern placed on each plate, no assumptions are made with respect to the reciprocity law, or with respect to whether the line intensities fall on the straight-line portion of the characteristic curve. Accurate analyses can be made only with clear plates, although attempts have been made to correct for background. None of these corrections have been found reliable.

The method described has several distinct advantages. Known standards need be used only once, that is, in making the working curve. Analyses can be made using a single exposure of the unknown. The several operations have been reduced to a laboratory routine so that the judgment of the observer need not be taken into account. No assumptions are necessary as to the validity of the reciprocity law, the introduction of intermittency effect, or in determining exact lengths of photographed lines. The accuracy of the method, with satisfactory unknowns, is about ±5 per cent. This will vary slightly for different elements and for different concentrations of the same element. A large number of elements can be analyzed at each exposure and at least ten exposures made on a single photographic plate. It is thus possible to obtain 50 or 60 analyses in a very short time on a single photographic plate. Analyses have been made in about 15 min., but with satisfactory apparatus this time can, without doubt, be shortened.

Acknowledgments:

The author wishes to thank the AC Spark Plug Co. for its financial support and for the alloys and chemical analyses made for these determinations. He also wishes to express his appreciation to Mr. D. W. Randolph, Director of Research at the AC Spark Plug Co., for his friendly cooperation during the progress of the experiments. Suggestions from Prof. O. S. Duffendack are gratefully acknowledged, and thanks are due to Dr. R. W. Smith for performing a large part of the experimental work.

DISCUSSION

Mr. M. L. Fuller.[1]—What is the form of electrode employed?

Mr. R. A. Wolfe.[2]—Usually we use metallic electrodes, but that is not necessary; we use them whenever possible to avoid carbon bands. However, most of our work is done in the region of a large spectrograph, between 3500 and 2500 Å, where carbon bands are not so serious.

Mr. Fuller.—What precision is experienced in your spectrographic analysis?

Mr. Wolfe.—On all the work we have done, such as the non-ferrous work reported upon, the precision is about ± 5 per cent. This accuracy can be increased by using more than one spectrogram; that is, by taking two or three spectrograms and averaging the results. For an accuracy of ± 5 per cent I am speaking of one single exposure.

Mr. Fuller.—Do you mean by that that if you analyze ten spectra the deviation from the mean would be less than 5 per cent?

Mr. Wolfe.—The deviation from the mean would be less than ± 5 per cent. Averaging several samples does two things: it takes care of slight non-homogeneities because it burns a larger sample; and if any errors have been made in reading the microphotometer, it averages out the inequalities, since a microphotometer has a certain least count within which one cannot read.

Mr. Fuller.—When you get up above 1 per cent, would not the precision of some of the chemical methods be better than the spectrographic method?

Mr. Wolfe.—Undoubtedly at the present time the precision of some chemical methods is better than the precision of the spectrographic methods, but when one considers how long we have used quantitative spectrographic methods, I would say that we are fast approaching the accuracy of chemical methods.

Mr. Fuller.—It should be emphasized that the spectrographic method is not to be applied to everything but only where it offers advantages over existing methods such as increased speed, increased precision, lower cost or ability to use a small sample.

Mr. Wolfe.—Results are sometimes incomplete and uncertain in chemical work, but that does not enter when one uses a spectrograph.

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[2] Research Physicist, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

Barium in nickel is tedious to get chemically; it takes 2 or 3 days; however one can do it in 12 to 15 min. with a spectrograph.

MR. EUGENE WAINER.[1]—For the past year and a half we have been analyzing ferrotitanium in a range of 15 to 25 per cent titanium and more recently titanium contents as high as 40 per cent, for titanium by spectrographic methods. The accuracy obtainable in these higher ranges usually is well within the ordinary limits of spectrographic accuracy, namely, ± 10 per cent. This figure is based on several hundred checks by standard wet methods as determined in our laboratory. The method has also been extended to other types of materials such as ores, glazes, and enamels; and the progress of the work thus far indicates that under proper conditions a like degree of accuracy is obtainable in the higher percentage ranges. In other words, we are applying spectrographic methods to the complete analysis of materials with a promising degree of success in the above mentioned limit of accuracy.

I believe that the restriction of the spectrographic method to low percentage ranges only is a mistake, and that in a large number of cases it will prove to be a valuable control instrument for high percentage ranges.

MR. W. R. BRODE.[2]—I feel that perhaps Mr. Wolfe has made some of us feel that it is necessary at all times to use a photographic plate which has been calibrated. Our experience in working with analyses in which we have not calibrated plates has been eminently satisfactory. For example, in a series of analyses for calcium and magnesium in fire-clay brick, which is about 75 per cent alumina and 25 per cent silica, and in which the amount of calcium and magnesium is less than 0.05 per cent, the chemical method is very difficult, but by the spectrographic method we have had highly satisfactory results by direct measurement of line intensities against our calibration curve through the use of the logarithmic sector and direct photometering.

MR. WOLFE.—I did not wish to convey that idea. The type of method we are using, however, absolutely necessitates the use of a calibrated plate and in the work we have done we have run up against a blank wall when we did not calibrate our plates. Other methods will work and sometimes may be better, but we have found this method to be very good, and without the calibration of the plates, we could not repeat our analyses with any degree of certainty. For special cases other methods have something to be said for them, but we have used this one very successfully.

MR. J. S. OWENS.[3]—It has been our experience that an intensity calibration pattern is necessary on each plate, not only to increase the accuracy, but also to make the method more satisfactory for routine use.

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[1] Spectroscopist, Titanium Alloy Manufacturing Co., Niagara Falls, N. Y.
[2] Associate Professor of Chemistry, Ohio State University, Columbus, Ohio.
[3] Research Investigator, X-ray and Spectroscopy Dept., The Dow Chemical Co., Midland, Mich.

I think that the use of the logarithmic sector and also the standards method may be made quite accurate in the hands of an experienced observer who is doing the same thing day in and day out, but our method, which is essentially the same as that described by Mr. Wolfe, is principally used as a commercial method to be used by any intelligent laboratory assistant, and for that purpose the calibrated plates are necessary and give better results.

MR. G. M. RASSWEILER.[1]—I should like to ask how you take into account, for routine work, the effect of impurities on the ratio of intensity of the two lines? Do you have to run a separate analysis?

MR. WOLFE.—The standard working curves are made from samples that contain the substances under investigation in about the same amount as the samples to be analyzed. Thus, a variation from 0.5 to 1.5 per cent of iron, or a variation from 1 to 5 per cent of manganese, would have no effect on the actual analysis as made from the combination, because the working curves are made from samples similar in composition to the substances being analyzed.

MR. RASSWEILER.—I understood you to say that even small amounts, amounts so small that you could not detect them spectroscopically, might have some effect on the ratio. If this is true it is difficult for me to see how you can depend on the results of spectrum analysis because even from your spectrum plate you could not determine whether or not foreign substances were present in sufficient amounts to affect the results.

MR. WOLFE.—I made that statement from the work we have done in gaseous discharges and investigations of that nature, knowing that a small amount of impurity will sometimes markedly affect the intensity of a given spectral line. I brought that out in the hope that we could get work done along that line to see if it did affect the same processes in metallurgical work and analyses of that type. I do not know that it does. I do know, however, that in the case I showed you with barium, nickel and copper, it does affect the analysis and that small impurities do show a large effect on the barium content.

MR. G. E. KUCK.[2]—Upon examining the curve presented in Fig. 1 of the paper it is apparent that the ratio of intensity of the chromium-nickel lines is an exponential function of the current. This proves to be the case for when the log $\frac{I_{Cr}}{I_{Ni}}$ is plotted against current (amperage) a straight line is obtained. Reference to this curve shows that equal variations in the current at 10 amp. and at 5 amp. give the same change in the log $\frac{I_{Cr}}{I_{Ni}}$. For example, a change of 0.2 amp. in the current at 10 amp. causes a change in the log $\frac{I_{Cr}}{I_{Ni}}$ from 9.800 to 9.855. At 5 amp. a similar fluctuation of 0.2 amp. causes a change in log $\frac{I_{Cr}}{I_{Ni}}$ from 0.400 to 0.455. In both cases the difference is 0.055.

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[1] Research Physicist, General Motors Research Laboratories, Detroit, Mich.
[2] Lucius Pitkin, Inc., New York City.

The plotting of log $\frac{I_{Cr}}{I_{Ni}}$ against current instead of $\frac{I_{Cr}}{I_{Ni}}$ is further justified when one examines the other curves in Mr. Wolfe's paper and finds that it is the log $\frac{I_x}{I_{Ni}}$ which is plotted against percentage and not $\frac{I_x}{I_{Ni}}$.

It is very interesting to know, as the author has demonstrated, how the ratio of intensity varies with a change in current. However, the author does not appear to have adequately proved the necessity of using 10 amp. instead of 8 or 6 when arcing nickel. It would seem more important to ascertain the actual variation in current when different amounts of current are used. From our experience it would appear that the least variation in current is obtained when 6 to 8 amp. are used, for lower and higher amperage produced certain conditions each of which in turn caused large variations in the current. These larger variations of current of course brought about larger variations in the relative intensities of the lines.

One might raise other interesting questions from the data presented in Fig. 1 of the paper. For example, when a current of 2 amp. is used, the intensity of the chromium line is six times that of the nickel line, but when a current of 12 amp. is used the nickel line is three times as intense as the chromium. Is this apparent reversal due to an increase in the intensity of the nickel line while the intensity of the chromium line remains constant? Or is it that the intensities of both increase but at different rates? Or, again, does the intensity of the chromium line decrease while the nickel line intensity remains constant or increases? Some very instructive points might be brought to light by the presentation of such data.

In their work with the logarithmic wedge sector Twyman and Harvey[1] plotted the log per cent of impurity against the difference in length of base metal and impurity line. Elsewhere Twyman and Simeon[2] proved, but did not stress, the fact that the difference in length of two such lines multiplied by some constant is equal to the logarithm of the ratio of intensity of the two lines. They would have obtained the same results by changing the difference in the length of line to the log ratio of intensity and plotting this against the log per cent. Following this procedure data taken from Figs. 2, 3, 4 and 5 of Mr. Wolfe's paper were plotted with the log per cent instead of per cent. It was found that within certain limits a straight line was obtained. The best agreement is shown by the magnesium-zinc curve (Fig. 3). Examination of both types of curve shows that the precision of the method is limited between certain percentages. For instance the determination of barium in nickel is limited to the range 0.02 to 0.16 per cent.

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[1] Twyman and Harvey, *Journal*, Iron and Steel Inst., Vol. 11 (1932).

[2] Twyman and Simeon, *Journal*, Optical Soc., Vol. 21, p. 169 (1930).

Apparently when amounts more than 0.16 per cent are determined the error becomes large.

MR. WOLFE.—Referring to Mr. Kuck's comments, I believe I mentioned in presenting the paper that all of the curves listed could be plotted on a semi-logarithmic scale and a straight line obtained. We have actually used this type of curve for several years in our own laboratory. The curves were drawn as shown to emphasize certain aspects of the sensitivity of the unknown in the alloys used.

I do not see how Mr. Kuck obtains the results given in the first paragraph of his questions. By referring to Fig. 1 in the paper it can be seen that at 5 amp. an arc current change of 0.2 amp. causes a change of $\frac{4}{10}$ of a log unit, in $\frac{I_{Cr}}{I_{Ni}}$ while at 10 amp., 0.2 amp. causes a change of less than $\frac{1}{10}$ of a log unit. This is even more noticeable at 2 amp. where a 0.2-amp. change causes a log unit change of over $\frac{5}{10}$.

Certainly one should operate the arc at a point of small sensitivity change for current fluctuations.

With a 2-amp. arc the conditions are such that the radiation of light from excited nickel and chromium atoms is a volume effect, there being very little absorption of resonance radiation. As the current through the arc is increased, a larger volume of the discharge results and absorption does take place. Since the nickel atoms are present in great percentages at all times, absorption takes place at a much smaller arc volume than for the comparatively few chromium atoms present. Consequently, as the arc volume increases the intensity of the nickel line remains nearly constant while the intensity of the chromium line decreases rapidly. Finally, since the arc volume increases only slightly above 15 amp., the ratio of $\frac{I_{Cr}}{I_{Ni}}$ changes very slowly.

Mr. Kuck calls attention to the fact that the curve of Fig. 4 is good for the analysis of barium in nickel only up to a barium concentration of 0.16 per cent. This is true for the particular conditions described. If higher concentrations of barium are present, other lines or other conditions of excitation are necessary and must be so selected. The general method described will be the same and can be made to fit any concentration of unknown material.

MR. C. C. NITCHIE.[1]—I am afraid some of those present who may not actually be working in this field, may get a rather wrong impression from some of the differences of opinion expressed at this meeting. I should like to point out that there is a large number of industrial laboratories using spectrographic methods, using them successfully and getting results to check their industrial operations and giving them valuable information. Most of the workers have been able successfully to work out methods that are adaptable to their particular problems.

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[1] Sales Engineer, Instrument Sales Division, Bausch & Lomb Optical Co., Rochester, N. Y.

REAR AXLE GEARS: FACTORS WHICH INFLUENCE THEIR LIFE

By J. O. Almen[1] and A. L. Boegehold[1]

Synopsis

Describes a series of endurance tests on rear axle gears which shows that gear tooth failures may be plotted on the familiar *S-N* curve when the gear stresses are properly calculated. Shows that the widely used Lewis formula for calculating gear tooth stress leads to serious errors. Also indicates that the chief benefit obtained from the use of alloy steels is the reduction in amount of distortion which results in lower stress concentration through more uniform tooth contact. Gives rotating-beam plain and notched-bar fatigue test results for several gear steels.

PART I—SPIRAL BEVEL REAR AXLE GEAR TESTS[2]

During the last seven or eight years, the General Motors Research Laboratory has run breakdown tests on some four hundred rear axle assemblies. The data obtained from so large a number of tests offered unusual opportunities for the study of actual machine elements as fatigue specimens in comparison with fatigue data from many sources on standard laboratory specimens. Of particular importance was the fact that fatigue tests on complete rear axle assemblies could be related to the actual service of these axles in the hands of many thousands of automobile owners through the service records maintained by the manufacturing divisions.

It is shown tenable to consider the gear tooth as a fatigue test specimen simulating an ordinary intermittently loaded cantilever beam with full tangential load carried on one tooth. As a result, many observations that have long puzzled engineers and metallurgists are found to fit in an orderly and rational manner with fundamental knowledge of design and materials.

It is further shown that axle gear failure in service is due to fatigue failure of the gear teeth under maximum low gear torque. Using this loading condition, the laboratory fatigue tests of the axle assembly rate the gears in the same order as in service.

Due to their influence on stress concentration, variations in design and manufacturing have a materially greater effect on gear life than do variations in materials and heat treatment. The effect of metallurgical

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[1] Head, Dynamics Section, and Metallurgist, Research Division, respectively, General Motors Corp., Detroit, Mich.

[2] Contributed by J. O. Almen.

Geo. L. Smith

change is obscured by the effect of these stress concentration factors, which include (1) elastic deflection of teeth, shafts and bearings, (2) surface cuts and scratches, (3) tooth shapes having abrupt change of section, (4) eccentric assemblies, (5) heat treatment distortion and incipient quenching and grinding cracks, and (6) non-uniform load distribution along the tooth.

It is suggested that to obtain improvement in gear life the engineer should first look to improvement in design and manufacturing before resorting to metallurgical change.

Axle Fatigue Testing Equipment:

The specialized type of testing equipment on which these tests have been run is shown in Fig. 1. The propellor shaft of the rear axle undergoing test is coupled to a large d.c. cradled dynamometer capable of delivering 1570 lb-ft. torque. The axle shafts of the rear axle are connected to two cradle-mounted a.c. power absorption units through a 13.66 to 1 speed increasing gear. This speed increasing gear is made from a commercial worm gear truck axle in which the worm wheel is used as the driving member and the worm is used as the driven member. Ten pole alternators are mounted directly on each of the worm shafts. During tests, the alternators are usually run at 725 r.p.m. which is synchronous speed with the alternating current supply to which they are connected. This not only provides simple speed control means but has the further advantage of reducing the electric current consumption through regeneration.

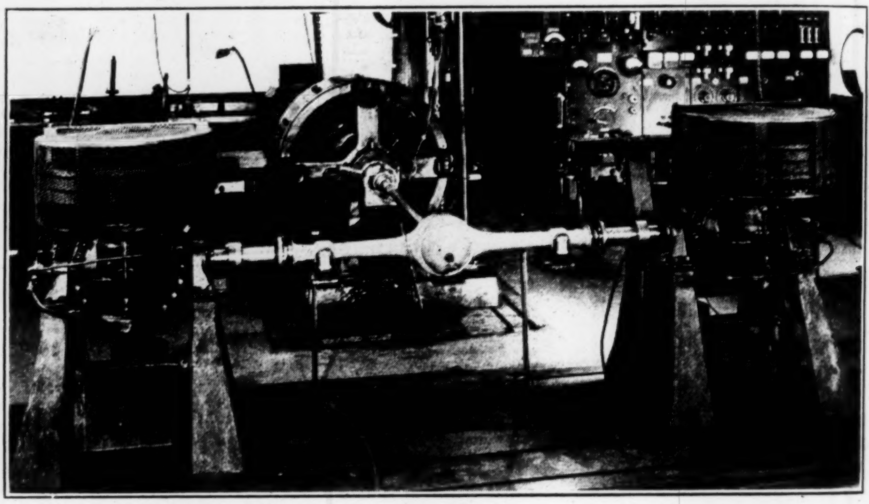

FIG. 1.—Axle Fatigue Testing Equipment.

The load applied to the axle gear during the test is usually equal to the maximum torque of the engine multiplied by the transmission low gear ratio, that is, approximately three times maximum engine torque. The test at this load is continued until failure occurs which, in practically all cases, is breakage of one or more pinion teeth. Records are kept of the propellor shaft load, axle shaft loads, oil temperature and the number of revolutions of the propellor shaft during the test. It is customary to run at least three axles of each design. The average duration of these runs is considered a measure of the relative merit of the axle for direct comparison purposes. As a fatigue test specimen, it is, of course, always better to use an average of several tests than to rely upon a single specimen. In the accompanying charts, each plotted point is, therefore, in most cases, an average of three or more axles.

Criterion for Axle Gear Failure:

It is rarely possible to devise a laboratory test of any structural part of an automobile that duplicates service conditions sufficiently to produce truly correlative test data. When all the test conditions are made faithfully to reproduce actual service conditions for the rear axle, it will be found that not only must complete vehicles be used but that they must include driving the vehicle under all conditions of roads and loads, including the type of driving that is usually considered abusive, such as driving through deep mud or sand, carrying large overloads, etc. It will not suffice that the test be conducted by a test driver since the proper proportions in various types of service will only be found through records of owner-driven cars. These data must be accumulated in the same manner as mortality tables are accumulated for the calculation of insurance rates with the difference that the automotive designer does not have the privilege of eliminating the poor risks, that is, the abusive driver.

At one time, it was generally believed that axle gear tooth failure in service was due to shock impact loading. In consequence, tests and materials specifications were in accordance with this theory of failure, which led the investigator astray, as discussed later in this paper.

Also, it was at one time suggested that axle gears might be overloaded due to impact loading under high-speed operation. This speculation in the case of axle gears was based on the general theory suggested by Buckingham[1] that gears in some types of high-speed service are subjected to high impact loads due to oscillation of the gears, and that gear fatigue failure is often due to the overstress resulting from such shock loads rather than to the normal driving loads.

To determine whether such impact loads were contributing to automobile rear axle failures, measurements were made of the elastic characteristics of the rear axle structure from the front end of the propellor shaft to the road tires. It was found that the elasticity in this driving train relative to the moment of inertia of the gears is such as to make impact loading of the type described by Buckingham practically impossible in automobile rear axle gears. Furthermore, the bulk of rear axle gear failures in service were found to originate in a few isolated sections of the country during the spring and fall seasons. Their geographical distribution and seasonal character indicated hard pulling rather than fast driving as the reason for failure. However, as a further check for impact loading, road tests were conducted with telephone receivers coupled across the gear teeth which showed continuous closed circuit under all driving conditions except when the torque was reversed.

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[1] Earl Buckingham, "Dynamic Loads on Gear Teeth," ***Research Publication***, Am. Soc. Mechanical Engrs. (1931).

In the beginning, the tests herein reported were conducted for the General Motors car divisions merely as routine durability, or breakdown, tests without any detailed consideration as to the manner of gear tooth failure or to the factors influencing this failure. The main purpose of the test was to establish the durability of a new design relative to a past design on which service experience data had been compiled. Initially, then, the procedure was simply a matter of orderly recording and study of test data. As the tests progressed, however, it became apparent that many popular conceptions of materials and design did not agree with test results. The formula in general use for calculating bending stress in gear teeth was found unreliable. No consistent difference could be found among the various steels and heat treatments used in production. As later analyzed, variations in stress concentration due to deflections, tooth forms, machining scratches, etc., had so great an effect on fatigue resistance as to obscure the effects of various alloys and heat treatments. Laboratory tests on standard specimens were not in quantitative, and few in qualitative, agreement with these tests on gears.

It must be emphasized that, at the time this type of breakdown test equipment was first put into operation, it was not generally recognized that normal pinion tooth failures in service were due to fatigue. Therefore, before the results obtained by this laboratory fatigue test would be admissible, it was necessary to establish that this test rated gear assemblies in the same order as these gear assemblies were rated in actual owner service. Furthermore, even if normal service failure was found to be due to fatigue, as was indicated by examples of failures showing typical fatigue fracture, it would still be necessary to find out how a laboratory fatigue test should be conducted to rate the gears in their proper order. That is, should the gears be run at relatively low loads and high speeds or should they be tested at high loads and low speeds? This question could only be answered by searching the service records for examples of production axles that were representative of several degrees of durability. The type of information sought for was found after reviewing thousands of service reports; that is, records were found showing relatively larger and smaller numbers of service failures for several production designs. With this information as a guide, it was found that axles tested under maximum low-gear torque on the axle testing equipment showed the same relative resistance to failure that was shown by the service records.

From these checks, it was believed reasonable to conclude that, when a rear axle gear fails in service, the failure is due to fatigue of the gear tooth as a result of intermittent loading; and that the fatigue life of a gear tooth is used up in the accumulative effects of short periods of operation under maximum low-gear torque.

From the comparison of the laboratory tests with service records, it was found that operation for 100,000 pinion cycles under maximum low-gear torque on the test equipment was equivalent to a lifetime of service in the automobile under the most severe operating conditions. The requirements for truck and bus axles are somewhat higher due to greater average severity of service.

In many machine elements, such as connecting rods, valve springs, crankshafts, etc., the number of maximum stress cycles occurring during the life of the machine is such a large proportion of total cycles that the permissible stress at the fatigue limit is the important value to be used in design. Many automobile parts, such as rear axle gears, axle shafts, transmission gears, etc., differ from ordinary machine elements in that the number of maximum stress cycles is such a small proportion of total cycles that they may be designed to operate under maximum loading at stresses far above the permissible stress at the endurance limit. As has been stated, the minimum required life of a rear axle pinion gear at maximum stress is only 100,000 cycles, equivalent to about 30 miles of road travel. The tooth stress in normal car operation, that is, in high gear, is so low as to be negligible from a fatigue standpoint.

Several cases are known of service failures of rear axle gears in automobiles in actual owner service, the entire history of which were known. On the average, the total number of pinion cycles of operation in the car was 1000 times the number of cycles run in the laboratory test. In other words, the severity of service in the hands of the hard driver is approximately one one-thousandth as great as the service in our laboratory test. With due qualification, it might be roughly stated that, in the car, there is only one maximum stress cycle per thousand total cycles.

As shown later, the maximum permissible design stress for automobile rear axle gear requirements, that is, the calculated stress equivalent to 100,000 cycles, is approximately 42,000 lb. per sq. in. as calculated by the modified stress formula. As a factor of safety, however, it is customary to design automobile rear axle gears to run not less than 250,000 cycles which is equivalent to a calculated stress of 37,000 lb. per sq. in. Note that this safety factor is small as compared to the several hundred per cent factor that experience has shown to be necessary in ordinary design practice in cases where fatigue data on the machine element itself are not available.

In such service as automobile rear axle gears, the permissible stress at the fatigue limit is not necessarily the best measure of material, and for similar reasons the usual tension tests may lead to the selection of inferior steel. The best material is that which when tested in a rear axle assembly will have the greatest resistance to fatigue in the region of 100,000 to 250,000 cycles.

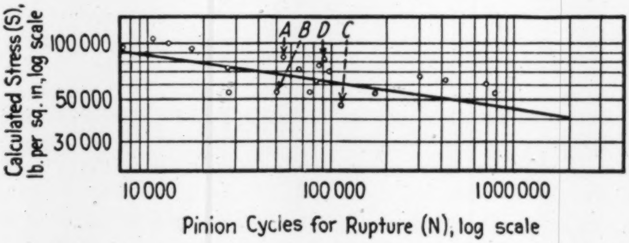


FIG. 2.—Spiral Bevel Gear Fatigue Curve Stress Calculated by "Lewis" Formula.

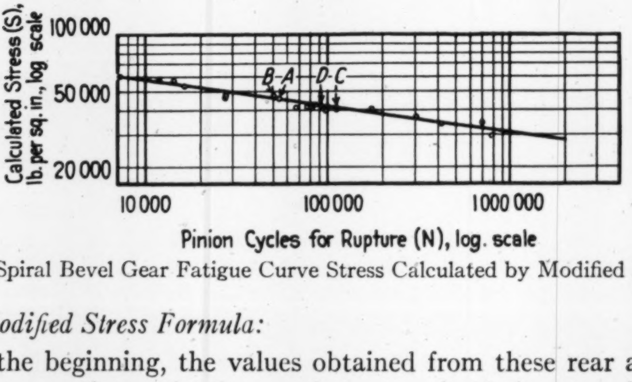


FIG. 3.—Spiral Bevel Gear Fatigue Curve Stress Calculated by Modified Formula.

Need for Modified Stress Formula:

From the beginning, the values obtained from these rear axle breakdown tests were plotted in the usual manner for fatigue specimen, as is shown in Fig. 2. In this log-log plot, the calculated stress of the specimen is shown on the vertical scale and the number of stress cycles before failure occurs is shown on the horizontal scale. Data points plotted in this manner should have lain reasonably close to a straight line if gear tooth failure were really due to fatigue. However, the plotting of the first 20 points, corresponding to 62 individual axles broken in test, resulted in wide scattering of the points as is shown in the figure. This scattering was most disconcerting since it indicated that gear failures were too erratic to permit their study as fatigue specimens unless some rational explanation for the scattering could be found. Among the reasons that might be accountable for the unusual scattering was possible errors in the method by which gear tooth stresses were calculated. The method that had been used was the gear tooth stress formula originally introduced by Dr. Wilfred Lewis some forty years ago.

A modification of the Lewis formula had been suggested by McMullen and Durkan in *Machinery*, June, 1922, but the new formula did not come into general use. When the tooth stress of the same 62 axle gears was recalculated by the McMullen and Durkan modification, they plotted to the curve shown in Fig. 3. Here we find little evidence of the disorder that characterized the original plot. The points lie as close to a straight line as could be expected from highly accurate laboratory fatigue specimens, notwithstanding the fact that these test points represent ordinary production axles of many sizes and designs. It is a striking proof of the greater accuracy of the modified gear tooth stress formula. Note that the gears represented by points *A* and *B* failed after approximately the same number of stress cycles, indicating that they were actually stressed alike. In Fig. 2, the gears represented by point *A* were calculated by the Lewis formula to be stressed 85,000 lb. per sq. in., whereas the gears represented by point *B* were stressed, according to the Lewis formula, to 55,000 lb. per sq. in. When calculated by the modified formula, these gears were found to be stressed alike, that is, approximately 47,000 lb. per sq. in. Or, comparing points *D* and *C*, Fig. 2, calculated by the Lewis formula, point *D* represent gears stressed to 85,000 lb. per sq. in. and point *C* gears stressed to 47,000 lb. per sq. in. By the modified formula and as plotted in Fig. 3, the stress was calculated at 43,000 lb. per sq. in. for point *D* and 41,000 lb. per sq. in. for point *C*. Thus, the Lewis method of calculating stress may introduce inaccuracies on the order of 80 per cent, whereas the McMullen and Durkan method reduces these inaccuracies to negligible amounts.

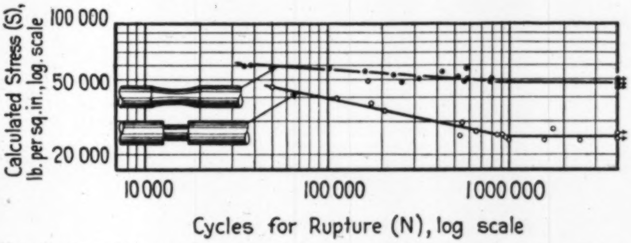


FIG. 4.—Effect of Shape of Specimen on *S–N* Diagram-Rotating Beam Tests.

In comparing Figs. 2 and 3, it will be noted that the stress scales in the two plots are quite different. No means are yet available for determining the actual stresses, and the stress scales used are, therefore, purely relative. Either stress scale may be multiplied by a constant without altering the real value of the plot.

Effect of Stress Concentration on Fatigue Life:

Figure 4 shows two fatigue curves taken from the University of Illinois *Bulletin No. 124* by H. F. Moore and J. B. Kommers entitled, "An Investigation of the Fatigue of Metals." The rotating-beam specimens from which these curves were made were identical as to material and heat treatment but different in form. The specimen for the upper curve, having an endurance limit at 49,000 lb. per sq. in. stress, is of the form shown on the chart; the lower curve showing an endurance limit of 24,000 lb. per sq. in. is for the shouldered specimen. This stress difference, however, is not real but is simply the result of calculating the stress in the shouldered specimen without allowance for the stress concentration resulting from the sudden change in section. The fatigue tests, which respond accurately to the real stresses, provide means for correcting the stress formula for the shouldered specimen in terms of the uniform stress specimen since the real stress is obviously the same for both forms.

The difference between the real stress and the calculated stress will not be the same for different forms of fatigue specimens; that is, the errors in stress calculation will vary depending upon local conditions producing stress concentrations. The true stress in any form of laboratory specimen having stress concentration can only be found, so far as the authors are aware, by a sufficient number of fatigue tests on the specimen to construct a fatigue curve from which the stress concentration factor may be determined by comparison with similar data on a simple specimen. This method of determining true stress is, unfortunately, not yet applicable to machine elements since it only admits of stress determination in terms of a uniformly stressed or other form of standardized specimen. The General Motors Research Division has attempted to correlate tests of rotating-beam fatigue specimens and rear axle gear tests with the hope that true stress could be determined and materials evaluated, but the results have been disappointing.

The Carburized Gear Tooth—Specifications for Case and Core:

The impact theory of tooth failure in axle gears has led to erroneous conclusions with regard to relative strength requirements of case and core. In consequence, laboratory tests designed to aid in evaluating materials have led the investigator astray, since these tests were responsible for the widely held belief that any carburized gear tooth should have a hard case and a tough core. The fatigue theory of failure alters the conception of case and core requirements.

For many years it was customary to test automobile gears in various impact machines. The designer of this type of test assumed that gear teeth in service were subjected to hammer-like blows, and he therefore attempted to duplicate in the laboratory this type of load. Under this test the best material was that which resisted the greatest number of blows notwithstanding the fact that the gear was usually ruined after the first impact. Hence, the specification that carburized gears must be of such materials and heat treatment as would produce a hard surface to resist wear and a tough core to resist breakage by impact.

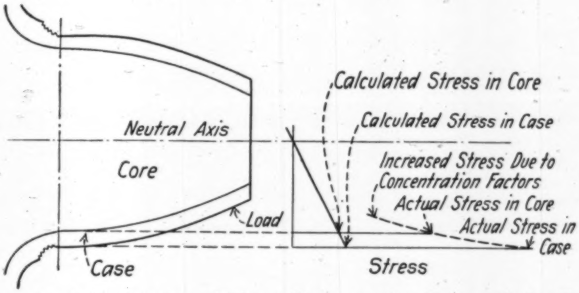

Calculated Stress in Core
Calculated Stress in Case
Neutral Axis
Core
Increased Stress Due to Concentration Factors
Actual Stress in Core
Actual Stress in Case
Load
Case
Stress

FIG. 5.—Distribution of Stress Between the Case and the Core.

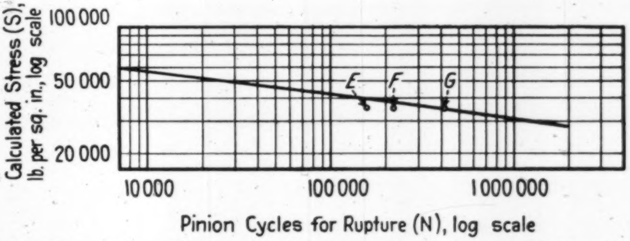


FIG. 6.—Spiral Bevel Gear Fatigue Curve Case Depth $E = 0.037$ in., $F = 0.045$ in., $G = 0.082$ in.

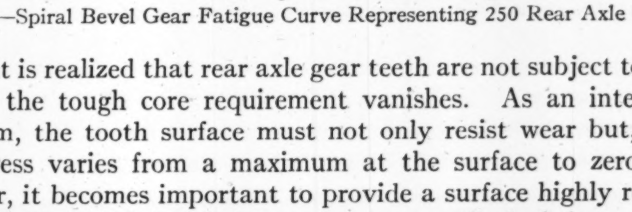


FIG. 7.—Spiral Bevel Gear Fatigue Curve Representing 250 Rear Axle Gears.

When it is realized that rear axle gear teeth are not subject to hammer-like blows, the tough core requirement vanishes. As an intermittently loaded beam, the tooth surface must not only resist wear but, since the bending stress varies from a maximum at the surface to zero near the tooth center, it becomes important to provide a surface highly resistant to fatigue in bending with less fatigue resistance required of the core depending upon the depth of carburization.

Figure 5 shows a qualitative distribution of stress between the case and the core. The vertical ordinate represents the half thickness of a gear tooth; the horizontal scale represents stress. The actual surface stress is usually far greater than the calculated value due to stress concentration caused by surface irregularities as is illustrated by the dotted line. As calculated, the maximum stress in the core is less than the stress in the case by a relatively small amount if the case is thin. Unless the fatigue resistance of the core is proportionally as great as the fatigue resistance of the case, fracture will start in the core. Stress concentration factors, such as rough or scratched surfaces, change of section, etc., have the effect of increasing the stress difference between the case and core, thereby reducing the fatigue strength requirement of the core below the value indicated by direct calculation.

Figure 6 shows the results of three groups of rear axle gears carburized to different depths. Point E is the average of three gears carburized to approximately 0.037-in. depth, point F is the average of three gears carburized to the normal depth of approximately 0.045-in. depth and point G is the average of three gears carburized to a depth of approximately 0.082 in. These data are too meager to be conclusive but they indicate that resistance to fatigue is perhaps improved with reasonable increase in depth of carburization. It should be noted that the normally carburized gears, point F, failed earlier than normal expectancy and that the deeply carburized gears, point G, about matched normal expectancy.

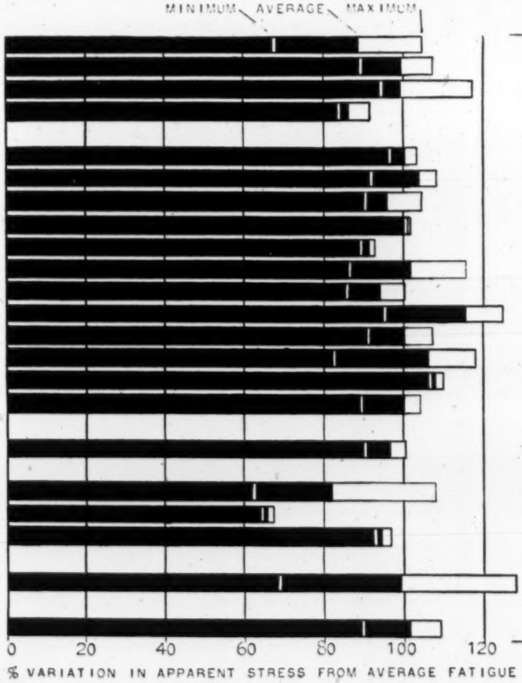
Effect of Material and Heat Treatment:

Notwithstanding the many metallurgical reasons and tests advanced to show that this or that alloy steel is best for rear axle gear purposes, there is no evidence from the present study to show that, among the alloys usually used for this purpose, one is superior to another. Figure 7 is the same average fatigue curve as Fig. 3 except that it records tests of 250 axles made from 22 combinations of alloys and heat treatments as noted in Table I. It will be seen that, regardless of material, heat treatment or grain size, the test points for any given material lie about equally divided above and below the average curve. Large deviations from the average are due to other reasons than metallurgical, as will be shown.

It is not intended to deny that there are real differences in the various alloys or in the effects of the various heat treatments. These differences, however, have been determined from rather ideal laboratory tests, under which conditions such differences are relatively large enough to become appreciable. In highly finished uniform-section structures, such as ball and roller bearings, wrist pins, ground shafts, and the like, the superior properties of expensive alloy steels are usually realized. In structures having high stress concentration, such as production rear axle gears, the

TABLE I.—VARIATIONS IN APPARENT STRESS FOR EQUAL FATIGUE LIFE FOR 248 REAR AXLES REPRESENTING 22 COMBINATIONS OF MATERIALS AND HEAT TREATMENTS.

The variations shown are due to design and manufacturing variables and not to material or heat treatment.



STEEL SPEC.	CARB. TEMP. °F.	CASE DEPTH INS.	QUENCH	REHEAT TEMP. °F.	QUENCH	DRAW TEMP. °F.	DRAW MINS.	NO. OF AXLES TESTED
2315-A	1660	.050-.055	IN OIL	1325-35	IN OIL	300	30	18
2315-A	1600	.045	IN OIL	1325-35	IN OIL	275	30	89
2315-A	1675	.050-.055	IN OIL	-	-	300	30	14
2315-A	1660	.040-.050	IN OIL	-	-	-	-	7
2515-A	1650	-	COOL IN BOX	1475	IN OIL	-	-	3
2515-A	1700	.050-.055	COOL IN BOX	1400	IN OIL	300	-	9
2515-A	1675	.045	COOL IN BOX	1380 CYN.	IN OIL	250	30	11
2515-A[a]	1675	.045	IN OIL	-	-	250	30	3
2515-A[a]	1675	.045	COOL IN BOX	1380 CYN.	IN OIL	250	30	2
2515-A	1675	.045	IN OIL	-	-	250	-	10
2515-A[a]	1675	.045	IN OIL	-	-	250	30	3
2515-A[b]	1675	.045	IN OIL	1380	CYN. FLASH & QUENCH IN OIL			2
2515-A[a]	1675	.045	TRANS. CYN. & REDUCE TEMP. TO 1350 & OIL QUENCH					12
2515-A	1700	.050-.055	COOL IN BOX	1400	IN OIL	300	-	12
2515-A	1660	.040-.050	IN OIL	-	-	-	-	3
2515-A	1675	.045	COOL IN BOX	1380	IN OIL	300	60	5
3115-A	1675	.050-.055	IN OIL	-	-	300	30	5
4615-A	1675	.050-.055	IN OIL	-	-	300	30	14
4615-A	1650-80	.050	IN OIL	1425	IN OIL	300	60	2
4615-A	1675	.040-.050	IN OIL	-	-	300	60	3
4620-A	1675	.040-.050	IN OIL	-	-	300	60	14
4815-A	1680	.050-.055	IN OIL	1420-30	IN OIL	300	30	7

[a] FINE GRAINED STEEL. [b] COARSE GRAINED STEEL.

properties of alloy steels, as determined by the usual laboratory tests, are not realized. The selection of steel for rear axle gears should be governed by warping tendencies, machining characteristics and cost.

In Table I are listed the twenty-two combinations of alloys and heat treatments represented by the test points shown in Fig. 7. The bar chart at the left of the table compares these combinations on a stress basis as indicated by the tests. Note that, on the basis of this comparison, coarse-grained 2515-A steel makes the best showing and that 4615-A steel is the poorest. This comparison, however, is not a true measure of the material or heat treatment. The differences are due to other factors as discussed below.

Figure 8 gives fatigue data for a series of tests designed to evaluate materials and heat treatments other than those used in production at that time. The series consisted of three axles each of nine combinations of materials and heat treatments. The early failure of these gears demonstrates the difficulty of producing good gears when made in small quantities due to the effects of unfamiliar warping characteristics of the steel. In preparing for tests of new production axle gears, it is customary to make a large number of gears and select from them the few that are good enough for use. The cutting is then adjusted to compensate for distortion. These tests are referred to in Table IV.

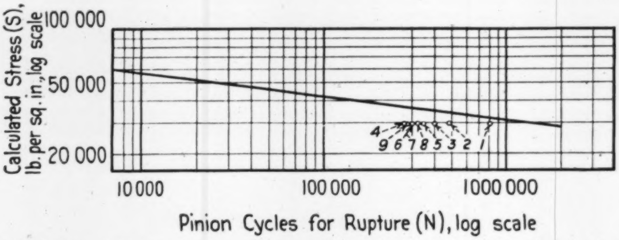


FIG. 8.—Spiral Bevel Gear Fatigue Curve Representing 27 Special Series Rear Axle Gears—See Table IV.

Effect of Stress Concentration Factors on Axle Life:

Properties of the alloys are obscured by the much greater effects of stress concentrations which, in gears, result from the tooth shape, machining scratches, deflection of the gears, shafts and bearings, eccentric assemblies, warping during heat treatment, etc. When considerable gain or loss is shown in the performance of a machine element, it is often ascribed to the particular alloy used when, in fact, it is probable that the gain or loss resulted from a change of one or more of the stress concentration factors.

Point *H*, Fig. 7, lies well above the average curve. This point is the average of three experimental rear axles that differed from production axles in that they were more rigidly supported and thus reduced the stress concentration due to deflection.

The gears represented by points *K*, *M* and *N*, Fig. 7, were of identical design, material and heat treatment, notwithstanding which their life varied as much as twenty to one. Point *K* is far above normal expectancy. The advantage in this case was reduced stress concentration due largely to smoother finish of the roots of the teeth. Point *M*, lying below the average, was the result of bad machining scratches, together with bad tooth contact, which increased the stress concentration. The gears representing the intermediate point *N* had good tooth contact as for *K* but severe machining scratches as for *M*.

Figure 9 shows the fractured pinion teeth from the axles represented by points *K* and *N*, Fig. 7. The one at the left was cut with slow cutter feed and the other with fast cutter feed. The difference in test durability was in the ratio of ten to one, that is, the coarse feed reduced the life of the gear from 1,000,000 cycles to 100,000 cycles. This test was not a deliberate test on the effect of cutter notches. The fine cutting feed was used while the gear cutting machine was being adjusted; the coarse feed was the normal cutting rate in production aggravated by the fact that the cutting blades were not of uniform height. The use of cutter blades of equal height and with corners rounded to the maximum permissible radius together with slower feed greatly reduced the stress concentration due to cutter scratches. Note, however, that the fractures in these gear teeth follow the cutter scratches in both cases, demonstrating that further increased durability may be expected if practical means for producing smoother finish can be found.

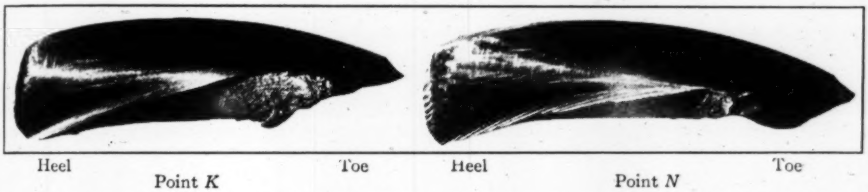


FIG. 9.—Fractured Pinion Teeth from Axles Represented by Points *K* and *N* in Fig. 7.

Figure 9 shows that these teeth were more highly stressed at one end than at the other since the characteristic fatigue fracture does not extend the entire length of the tooth. Failure started at the root of the heel (large end) of the tooth and moved radially outward at that point as the fracture progressed toward the toe (small end) of the tooth. This is typical of all gear failures in our tests and is mainly the result of elastic deformations, principally in the pinion anti-friction bearings, causing load concentration at the heel of the tooth, as discussed later.

The following table gives data on axles represented by points *O*, *P*, *R*, *S*, *T*, *U* and *W*, Fig. 7:

Plot Point	Number of Tests	Steel Specifications	Minimum Life	Maximum Life	Average Life	Average Life, Percentage of Expected Life
O..................	2	4615-A	656 700	969 000	813 000	4.8
P..................	5	4615-A	556 000	2 072 000	1 178 000	7.0
R..................	4	2315-A	1 120 000	1 700 000	1 422 000	8.4
S..................	2	2315-A	141 000	242 000	191 600	19.0
T..................	3	4615-A	233 000	537 000	427 600	43.0
U..................	3	2315-A	218 500	1 275 000	604 000	60.0
W..................	3	4615-A	195 500	450 800	293 000	122.0

Points *O*, *P* and *R* represent axle gears that were unusually large for the car in which they were used, having, by extrapolation, a normal expectancy of 20,000,000 cycles, or two hundred times the minimum requirement. Because of their size, these gears were safe notwithstanding extremely high stress concentration factors, and there was, therefore, no great incentive to improve their performance. The gears represented by points *S*, *T* and *U* were for the same make of car but of somewhat smaller relative size. Although still over-size, an improvement was made in the rigidity of the pinion bearing which is reflected in the position of the test points relative to the average curve. Subsequent increase of engine size required further improvement in design and shop practice with the result that these smaller gears now have somewhat more than normal expectancy, as given by point *W*. The two gears represented by point *O* are responsible for the poor showing of 4615-A steel in Table I which, as has been explained, was not the fault of either material or heat treatment.

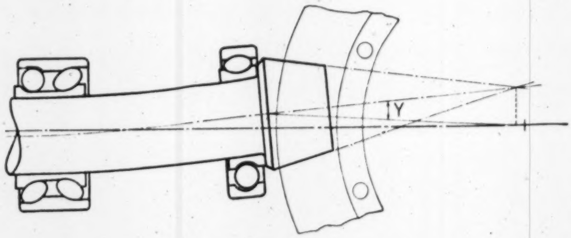


FIG. 10.—Exaggerated Sketch of Deflection of a Pinion Under Load.

Point *V*, Fig. 7, represents the average of seven axle gears of identical design, material and heat treatment with gears that formerly had given normal expectancy. This test point was low because the manufacturing had been transferred to a new plant and new personnel and the shop technique had not yet been mastered. Later gears of this design and material returned to the normal curve.

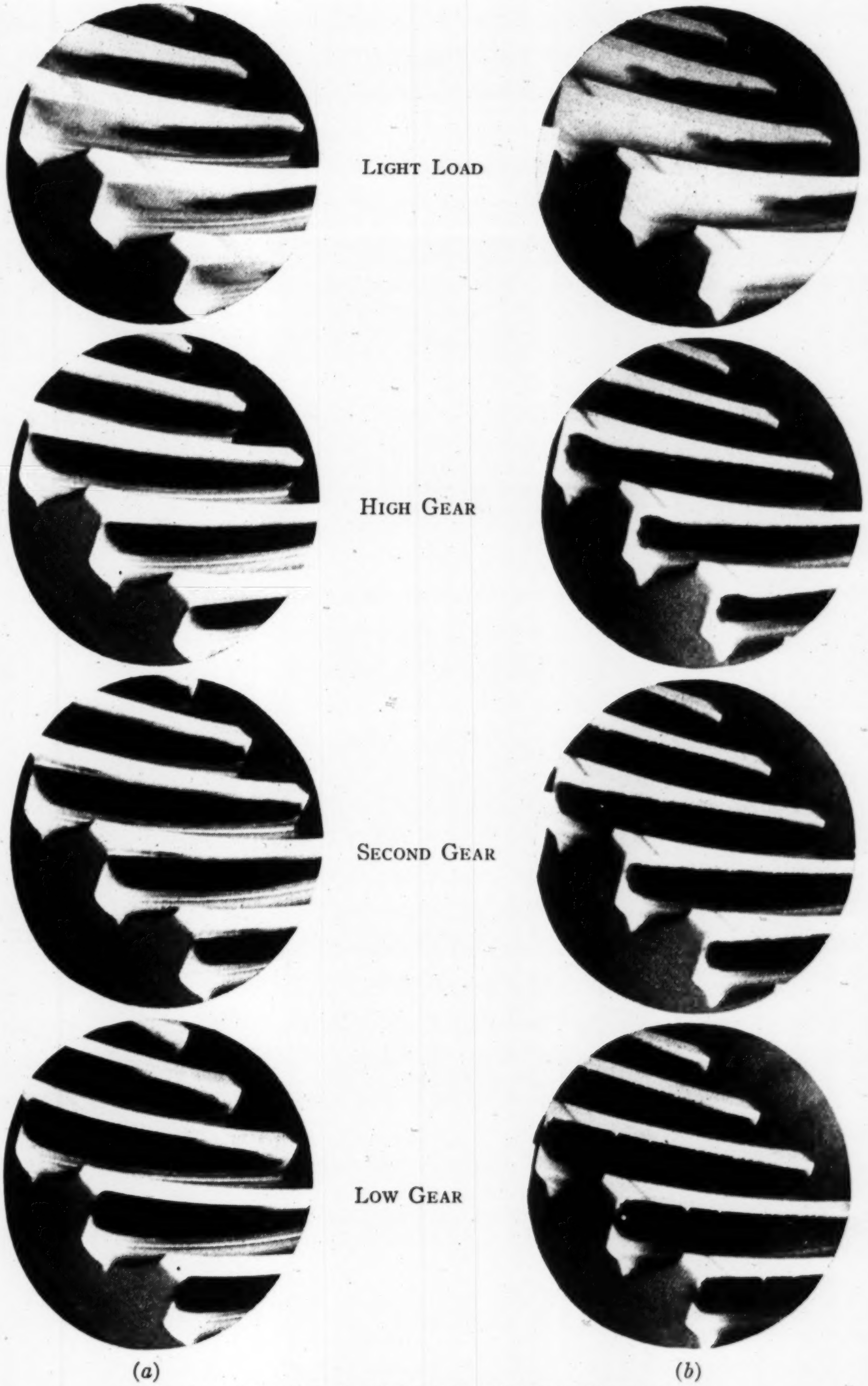

LIGHT LOAD
HIGH GEAR
SECOND GEAR
LOW GEAR
(a)
(b)

FIG. 11.—Photographs of Gears Made Under Four Loads.

Factors Affecting Load Concentration at the Heel of the Tooth:

Figure 10 shows an exaggerated sketch of deflection of a pinion under load and the stress concentrating effect of such deflection on the contracting teeth. Theoretically, the apex of the pinion cone coincides with the cone apex of the gear. This ideal condition does not prevail in practice inasmuch as all parts of the gear assembly are deformed when load is applied. The pinion bearing deformations are such as to shift the pinion axis through an angle which tends to localize the tooth loads at the heel of the teeth. A measure of this concentration is the angle Y, Fig. 10. Deflections in other directions are less serious in effect since they do not result in as serious shifting of the load lengthwise of the teeth.

A part of normal routine in our gear testing is the photographing of the tooth contact patterns of all gears tested. The photographs are taken while gears are running at constant speed under constant load by means of a neon lamp stroboscope. The rear axle is installed as is shown in Fig. 1 with the back cover plate removed. The axle is brought up to speed and load. A neon lamp, of special form, is flashed in time with the gear teeth by means of a cam on the input dynamometer which has as many lobes as the pinion has teeth, with the result that the gears appear to be stationary. The teeth are then sprayed with a specially prepared, quick-drying paint which dries before reaching the point where the gears are in mesh. This paint must not only cover the teeth and dry quickly but must also possess lubricating qualities since the gears must be free from oil during the photographing process. As the ring gear teeth come into contact with the teeth of the mating pinion, the paint is wiped off in the contact region giving sufficient color contrast to enable pictures being taken with an ordinary camera. As shown in Fig. 11 pictures are made under four loads corresponding to a light driving load, full engine torque in direct drive, full engine torque in second gear, and full engine torque through low gear. Note that at light load the gears made contacts well toward the small ends of the teeth and that as the load was increased the contacts shifted toward the large ends of the teeth and away from the small ends. This is as would be expected from deflections of the kind shown in Fig. 10 and from the fractures shown in Fig. 9.

Figure 11 (*b*) shows a set of tooth contact photographs made from a gear identical in every respect with the gear shown in Fig. 11 (*a*) except that the gear cutting machine was adjusted to give greater toe contact. Note that the shifting of contact with increasing load is less severe in Fig. 11 (*b*) than in Fig. 11 (*a*). This is most apparent when comparing the light load photographs and the highest load photographs. The reduction in stress concentration following the altered machine setting increased the life of the gear from 104,000 to 264,000 cycles.

Reduction of load concentration by setting the machine to cut toe contact is limited by the tendency of the gear to be noisy. Partial compensation for the inevitable shifting of contact with load is accomplished by cutting the teeth of the pinion to a spiral curve that differs from the spiral curve of the mating gear as is shown in Fig. 12. The smaller curvature of the pinion tooth curve permits this tooth to rock on the greater curvature of the gear tooth as the angular deformation varies. If these radii were equal, it is evident that the slightest angular deformation would shift the load from one end of the tooth to the other with consequent high stress concentration. The greater the angular deflection, the greater must be the difference in radii of the teeth of the two mating gears but this also leads to greater load concentration since it limits the useful length of the teeth. Obviously, improvement would follow reduction in angular deflection permitting less difference in the radii of the teeth and thus producing more uniform stress distribution.

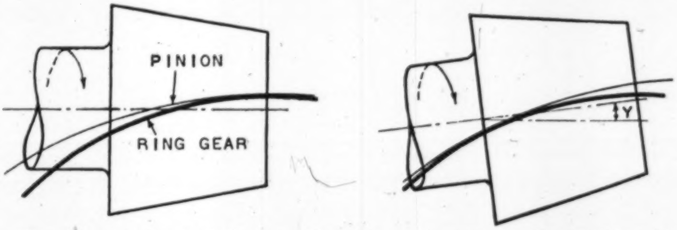


FIG. 12.—Showing Method of Cutting Teeth to Compensate for Shifting of Contact.

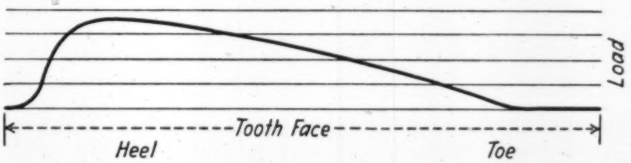


FIG. 13.—Distribution of Load Along the Pitch Line of the Pinion Tooth.

The photographs of the type shown in Fig. 11 are satisfactory for routine estimates of deflections and resulting load concentrations. They are quickly made and easily interpreted and have supplanted the old method of deflection measurements by indicators in the General Motors Research Laboratory. This method, however, does not permit quantitative measures of load distribution. For this purpose, in one case, a series of etching tests was made by applying a static load equal to second gear torque on an assembled axle and etching the surfaces of the gear teeth by passing sulfur dioxide and water vapor through the axle housing for a period of 30 hr. The areas of the gear teeth in actual contact were not etched and it was, therefore, possible to measure the extent of the contacting areas. From these measurements and the known curvature of the teeth, it was possible to calculate the actual compressive stresses and the distribution of load, which were checked against the applied load and found to agree within about 10 per cent. The tests were repeated a sufficient number of times with slight changes in the phase relationship of the gears to construct a plot of load distribution over the face of the teeth.

Figure 13 shows, as a composite of the loads on the three teeth in simultaneous contact, the qualitative distribution of load along the pitch line of the pinion tooth. The load concentration at the heel (large end) of the tooth is clearly indicated; likewise, the fallacy of rating gear tooth loads in terms of the length of the tooth face. The biased distribution of load would be even more pronounced under maximum low-gear torque.

Stress Distribution Between Pinion and Ring Gear:

An important consideration in the design of spiral bevel gears is the proper proportioning of stress between the two mating gears. As stated previously, the permissible calculated stress in the pinion teeth has been found to be approximately 42,000 lb. per sq. in., equivalent to 100,000 stress cycles. The permissible calculated stress in the ring gear has, by these same tests, been found to be 62,000 lb. per sq. in. With this stress distribution, either the pinion or ring gear would be liable to failure by fatigue. In part, the higher permissible ring gear stress is due to the fact that automobile rear axle gear ratios are on the order of four to one and, therefore, each ring gear tooth is stressed only one-fourth as often as the pinion tooth. The required minimum life of the ring gear, therefore, need be only 25,000 stress cycles, which corresponds to approximately 51,000 lb. per sq. in. (see Fig. 3). The balance of the permissible stress discrepancy, that is, the difference between 51,000 lb. per sq. in. and 62,000 lb. per sq. in. appears to be due to lower stress concentration in the ring gear teeth, probably as a result of less severe cutter scratches.

Pitting and Scoring:

In addition to tooth breakage, two other forms of gear tooth failure are recognized, namely, pitting and scoring.

Pitting has been found to be fatigue failure of the surfaces of the teeth due to compressive stresses. This stress is calculated by the method introduced by Hertz and reaches values on the order of 400,000 lb. per sq. in. maximum in the contact area as determined by the etching tests described above. Pitting has been developed in laboratory tests but has not yet been properly related to service. It is not now sufficiently serious in service to require immediate attention. All anti-friction bearings are subject to failure by pitting and are, therefore, made of steels having high resistance to compressive stress fatigue.

Scoring is the destruction of the tooth surface by tearing of the metal. The indications are that actual welding of small areas of the contacting teeth occurs under high unit pressures and the heat of friction, and that these small welded particles are responsible for the scoring. Among the factors known to influence scoring is the surface hardness of the teeth. The standard penetration tests are unsuitable for measuring surface hardness of the carburized gear tooth. Scoring usually occurs at high speed and, therefore, not at the highest unit pressures imposed on the gear teeth. The product of unit pressure and sliding velocity at maximum car speed in high gear is a fair measure of scoring tendency. With ordinary mineral oil, a $P\ V$ value of 1,500,000 is permissible for spiral bevel gears where P = compressive stress (Hertz maximum contact stress) in pounds per square inch and V = sliding velocity in feet per second. This $P\ V$ value may be greatly increased by the use of extreme pressure lubricants.

CONCLUSIONS

In supplementing the conclusions noted at the beginning of this paper, it appears in order to emphasize the following points.

For better utilization of material in machine elements the engineer should first look for possible improvements in design and shop practice before resorting to metallurgical change. The engineer prepares his designs without giving sufficient thought to the stress concentration factors noted herein. His designs often appear to assume that the materials are rigid. Axle bearings are usually selected for their load-carrying capacity rather than for their rigidity with the result that the gears are heavier than necessary.

The production man is usually willing to cooperate with the engineer when he is appraised of the detrimental effects of certain shop practices.

Laboratory tests of standard test specimens are often interpreted as being directly applicable to machine elements without regard to the wide differences in form, finish and type of loading. In the process of manufacture, the machine element may be so altered by surface decarburization, for example, that it does not represent the material of the test specimen.

When laboratory tests are made on actual machine elements, sufficient care is not always taken to assure that they agree with the results obtained in service. Such tests are often more misleading than specimen tests, since the results are more readily accepted at their face value. Reliable testing technique can only be developed by reproducing in the laboratory the type of failure and order of merit encountered in the field.

Automobile engineers and metallurgists have access to service data. These records, intelligently used, provide means whereby laboratory tests on machine elements may be related to service as in the present study of axle gears.

PART II—REAR AXLE GEAR STEEL TESTS[1]

Part I of this paper has shown how the results of rear axle fatigue tests made on a rear axle dynamometer correspond with service data. However, using complete sets of rear axle gears as test specimens is a rather expensive way of investigating a large number of gear steels and heat treatments. It, therefore, seemed reasonable that an investigation of the physical properties of gear steels might be a less expensive way of finding the best gear steel.

Since a gear tooth is subjected to bending stresses, it was reasoned that a bending test should give the desired information. At first, it was considered unnecessary to conduct fatigue tests, in spite of the fact that gear teeth are subjected to repeated stress applications, because so many investigators have already established the general relationship between ultimate strength and fatigue endurance limit. But since resistance to shock is usually considered a desirable property in steel parts of high hardness, it was decided to determine this property along with the bending tests.

TABLE II.—CHEMICAL COMPOSITION OF STEELS INVESTIGATED.

Tests Involved	G.M.C. Specification Number	Chemical Composition, per cent							Grain Size, A.S.T.M. Classification
		Carbon	Manganese	Phosphorus	Sulfur	Nickel	Chromium	Molybdenum	
Flexure tests—fatigue tests on standard specimens	2315	0.15	0.54	under 0.025	0.021	3.68	....	....	2 to 5
Flexure tests—fatigue tests on notched specimens	2315	0.18	0.48	under 0.025	0.017	3.58	....	....	6
Rear axle gear tests other than production treatment	2315	0.17	0.43	under 0.025	0.023	3.64	....	....	6 to 7
Rear axle gear tests—production treatment	2315	0.18	0.56	under 0.025	0.027	3.65	....	....	4
Flexure tests—fatigue tests on standard specimens—fatigue tests on notched specimens—rear axle gear tests	2330	0.31	0.70	0.023	0.023	3.31	....	....	5 to 7
Flexure tests—fatigue tests on standard specimens—fatigue tests on notched specimens	2512	0.12	0.47	0.020	0.016	4.90	....	....	6
Flexure tests—rear axle gear tests	4815	0.15	0.49	under 0.025	0.011	3.44	....	0.34	6
Flexure tests—fatigue tests on notched specimens—rear axle gear tests	MnMo	0.17	1.75	0.018	0.021	....	....	0.23	7
Flexure tests—fatigue tests on notched specimens—rear axle gear tests	1315	0.15	2.14	under 0.025	0.023	....	....	....	2 to 4
Flexure tests	3130	0.31	0.64	0.020	0.021	1.14	0.70	....	..
Fatigue tests on notched specimens	4620	0.23	0.69	under 0.025	0.018	1.66	....	0.33	7
Fatigue tests on notched specimens	3115	0.14	0.49	under 0.025	0.026	1.08	0.71	....	6

The preparation of test specimens and details of test procedure are given in an appendix to this paper.

Table II gives the chemical analysis and grain size of all the steels tested in this investigation.

Table III gives the hardness, impact, and flexure test results obtained.

The treatment giving the best combination of physical properties for each steel was selected for further investigation with gears subjected to a rear axle test.

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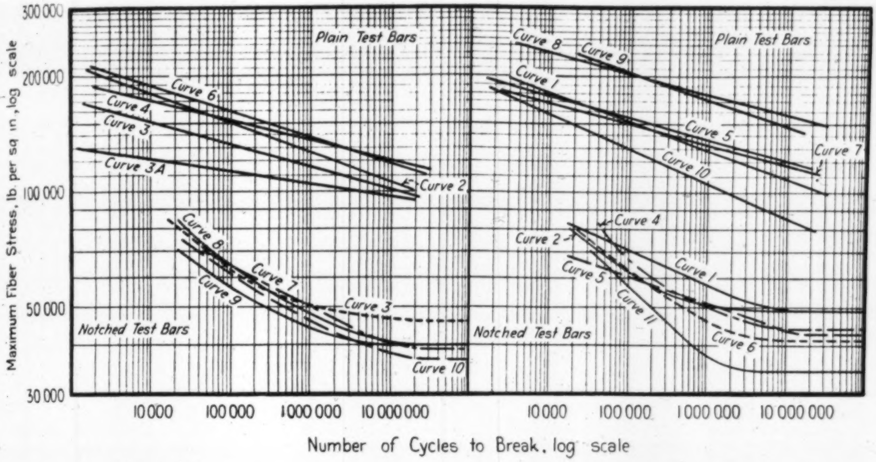
[1] Contributed by A. L. Boegehold.

Rear axle ring gears and drive pinions were made up of each of the steels and subjected to the heat treatments selected. Great care was observed in matching ring gears and pinions to obtain satisfactory contact. All of the sets finally subjected to test were passed by the inspector in charge of production of this job, even though the tooth contacts were not quite as good as regular production standards. The rear axle fatigue test



PLAIN TEST BARS

Curve 1. 2315—Carburized 6 hr. at 1650 F. Oil quenched from carburizing heat—drawn at 300 F.

Curve 2. 2315—Carburized 6 hr. at 1650 F. Transferred from carburizing heat to furnace at 1375 F., held until bars reached furnace temperature. Oil quenched—drawn at 300 F.

Curve 3. 2315—Carburized at 1650 F. Quenched in oil from carburizing heat. Reheated in furnace to 1330 F. drawn at 300 F.—0.042-in. case.

Curve 3A. 2315—Treatment same as No. 3—0.032-in. case.

Curve 4. 2315—Carburized 6 hr. at 1650 F. Quenched in oil from carburizing heat. Reheated in furnace to 1475 F. Oil quenched—drawn at 300 F.

Curve 5. 2315—Carburized 6 hr. at 1650 F. Cooled in box in furnace. Reheated in furnace to 1475 F. Oil quenched—drawn at 300 F.

Curve 6. 2315—Carburized 6 hr. at 1650 F. Oil quenched. Surface finish polished. Reheated in cyanide to 1475 F. Oil quenched—drawn at 300 F.

Curve 7. 2330—Carburized 3 hr. at 1650 F. Transferred from carburizing heat to furnace at 1375 F. Held until bars reached furnace heat. Oil quenched—drawn at 300 F.

Curve 8. 2330—Carburized 6 hr. at 1650 F. Oil quenched. Reheated in furnace to 1375 F. Oil quenched—drawn at 300 F.

Curve 9. 2330—Treatment same as Curve 8, except carburized for 3 hr. at 1650 F.

Curve 10. 2512—Carburized 6 hr. at 1650 F. Transferred from carburizing heat to cyanide at 1350 F. Held 10 min. Oil quenched—drawn at 300 F.

NOTCHED TEST BARS

Curve 1. 4620—Carburized 8 hr. at 1675 F. Oil quenched.

Curve 2. 4620—Carburized 8 hr. at 1675 F. Transferred from carburizing heat to cyanide at 1400 F. Held until bars reached temperature of bath. Oil quenched.

Curve 3. 3115—Carburized 8 hr. at 1650 F. Oil quenched. Reheated in furnace to 1475 F. Oil quenched—drawn at 300 F.

Curve 4. 2512—Carburized 8 hr. at 1650 F. Oil quenched—drawn at 300 F.

Curve 5. 2330—Carburized 4 hr. at 1650 F. Oil quenched. Reheated in furnace to 1375 F. Oil quenched—drawn at 300 F.

Curve 6. 2315—Carburized 8 hr. at 1650 F. Oil quenched—drawn at 300 F.

Curve 7. 2315—Carburized 8 hr. at 1650 F. Transferred to furnace at 1375 F. Held until bars reached furnace heat. Oil quenched—drawn at 300 F.

Curve 8. 3115—Carburized 8 hr. at 1650 F. Oil quenched—drawn at 300 F.

Curve 9. 2315—Carburized 8 hr. at 1650 F. Oil quenched. Reheated in furnace to 1330 F. Oil quenched—drawn at 300 F.

Curve 10. 1315—Carburized 8 hr. at 1650 F. Oil quenched—drawn at 300 F.

Curve 11. 1.75 per cent MnMo—Carburized 8 hr. at 1650 F. Oil quenched—drawn at 300 F

FIG. 14.—Rotating-Beam Fatigue Tests of Notched and Plain Bars Compared.

was run with a calculated stress of 29,130 lb. per sq. in. imposed on the pinion tooth.

The results of rear axle tests are given in Table IV. The life of gears in rear axle endurance testing was unrelated to physical properties as determined by flexure tests or by impact tests. What does such a result signify? We concluded that there were several possible explanations that would have to be investigated:

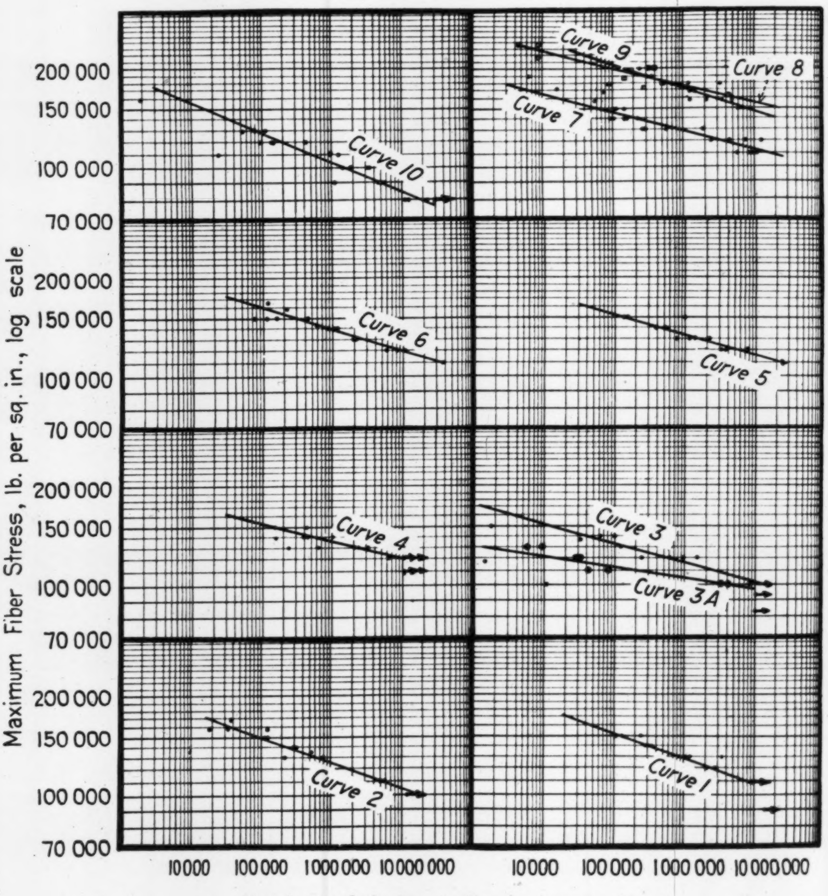


FIG. 15.—Rotating-Beam Fatigue Tests of Plain Bars.

1. The resistance to failure from repeated loading does not always bear the same relation to the flexural strength in different steels and treatments.

2. The effect of stress concentration as the result of tool marks at

the base of teeth and also due to the section change at the base of teeth was greater for some steels and treatments than for others.

3. The nature of tooth contacts as a consequence of distortion of the gears in heat treatment or due to excessive deflection from mountings caused higher unit stresses in some gear teeth than in others.

To determine whether the first was true, rotating-beam fatigue tests were made, the results of which are summarized in Fig. 14. Individual curves showing each point, to show the amount of variation of individual determinations from the plotted curve, are shown in Fig. 15.

It will be noted that many of the points indicate delayed fracture after 10,000,000 cycles of stress, an occurrence previously reported by French in fully hardened materials comparable with the ones under discussion.[1]

Table V gives a comparison between results from flexure tests and from rotating-beam fatigue tests. It will be noted that the flexural strengths were determined on bars carburized 8 and 4 hr. while the fatigue tests were made on specimens carburized 6 and 3 hr., respectively. This comparison is permissible because the flexural strength is not appreciably affected by case depth as will be noted in Table III. The results in Table V show that the first hypothesis is true in that the resistance to repeated loading for various steels and heat treatments does not always bear the same relation to the flexural strength. The ratio of endurance limit to flexural strength however does not vary sufficiently to explain the lack of agreement between rear axle test life and flexural tests because rear axle test life is not indicated by results of rotating-beam fatigue tests on standard fatigue test specimens. "Broadly speaking," as Gillett has stated,[2] "the ratio of endurance limit to tensile strength is unaffected by the kind of alloy or heat treatment used to obtain the tensile strength." This conclusion however does not necessarily apply under conditions of stress concentration.

On the basis of calculation according to the modified Lewis formula, the stress in the drive pinion tested was 29,130 lb. per sq. in. The approximate life of gears of regular production material and heat treatment, in rear axle tests subjected to low gear torque is 800,000 load applications. A fatigue test specimen of the same material and subjected to the same heat treatment requires the imposition of a stress of 117,000 lb. per sq. in. to cause failure in 800,000 cycles (see Fig. 15). Are we to conclude from this that the actual stress induced in the gear tooth was 117,000 lb. per sq. in.? If so the difference between 29,130 and 117,000 lb. per sq. in. represents the additional stress imposed on the gear tooth not accounted for by calculations. Since the gear tooth in the rear axle test is stressed only in tension on the drive side of the tooth, comparison should really

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[1] H. J. French, "Fatigue and Hardening of Steels," *Transactions*, Am. Soc. Steel Treating, Vol. 21, October, 1933, pp. 899–946.

[2] H. W. Gillett, "Effect of Alloying and Heat Treatment on the Endurance Limit of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 291 (1930).

TABLE III.—HARDNESS, IMPACT AND FLEXURE TEST RESULTS.

Treatment After Carburizing	Carburized at 1650 F., hr.	Case Depth, in.	Surface Hardness, Rockwell "C" Scale	Core Hardness, Brinell	Maximum Fiber Stress, lb. per sq. in.		Charpy Impact, ft-lb.
					Proportional Limit	Ultimate	
2315 STEEL							
Quenched from carburizing heat. Drawn at at 300 F.	4	0.041	59.0 to 61.0	302	158 300	292 000	99
	8	0.053	60.5 to 62.5	363	141 700	288 000	95
Transferred from carburizing box at 1650 F. to furnace at 1375 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F.	4	.....	60.0 to 61.5	341	173 000	300 500	129
	8	.....	59.0 to 59.5	341	149 000	293 000	100
Quenched from carburizing heat. Reheated to 1330 F. Quenched and drawn at 300 F. — fine grained	..	.....	....	....	144 000	196 000	101
	8	.....	63.0 to 64.0	187	153 600	217 900	152
coarse grained	4	.....	60.0 to 60.5	197	129 600	197 700	120
	8	.....	59.0 to 60.5	187	129 600	187 200	90
Quenched from carburizing heat. Reheated to 1475 F. Quenched and drawn at 300 F.	4	.....	60.0 to 61.0	341	173 000	305 000	....
	8	.....	60.0 to 60.5	341	187 000	296 000	104
2330 STEEL							
Transferred from carburizing heat to furnace at 1450 F. Held until bar reached temperature of furnace. Quenched and drawn at 300 F.	1	0.012	59.0	514	214 000	283 000	
	2	0.022	62.5 to 63.0	514	195 000	259 000	
	3	0.030	61.5 to 62.0	477	182 300	293 000	
	4	0.035	59.0 to 60.0	514	168 000	259 500	
	8	0.050	59.0 to 59.5	514	240 000	259 500	
Transferred from carburizing heat to furnace at 1375 F. Held until bar reached temperature of furnace. Quenched and drawn at 300 F.	2	0.022	62.5 to 63.0	514	216 000	298 000	
	3	0.030	63.0 to 63.5	514	197 000	288 000	
	4	0.035	61.0 to 61.5	514	173 000	266 000	
	8	0.050	61.5 to 62.5	277	278 500	307 500	
2330 STEEL							
Quenched from carburizing heat. Drawn at 300 F.	4	0.035	59.5 to 60.0	514	197 000	197 000	28
	8	0.050	58.5 to 59.0	514	206 400	206 400	23
Transferred from carburizing box at 1650 F. to furnace at 1450 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F.	4	.....	59.0 to 60.0	514	168 000	259 500	18
	8	.....	59.0 to 59.5	514	240 000	259 500	28
Transferred from carburizing box at 1650 F. to furnace at 1375 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F.	4	.....	61.0 to 61.5	514	173 000	266 000	18
	8	.....	61.5 to 62.5	477	278 500	307 500	30
Quenched from carburizing heat. Reheated to 1330 F. Quenched and drawn at 300 F.	4	.....	62.5 to 63.0	321	142 000	206 400	47
	8	.....	61.0 to 63.0	269	193 000	216 000	12
Quenched from carburizing heat. Reheated to 1375 F. Quenched and drawn at 300 F.	4	.....	62.5 to 63.0	514	240 000	336 000	30
	8	.....	60.0 to 61.5	495	264 000	322 000	22
Quenched from carburizing heat. Reheated to 1475 F. Quenched and drawn at 300 F.	4	.....	61.5 to 62.0	514	268 000	293 000	21
	8	.....	61.0 to 62.0	514	230 500	230 500	35
3½ PER CENT NICKEL-MOLYBDENUM STEEL							
Quenched from carburizing heat. Drawn at 300 F.	4	0.041	61.0 to 62.0	388	180 000	316 000	149
	8	0.057	60.0 to 61.0	363	173 000	283 000	92
	8	.....	60.0 to 61.0	388	189 600	290 880	84
Transferred from carburizing box at 1650 F. to furnace at 1375 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F.	4	.....	62.5 to 63.0	388	163 000	313 000	143
	8	.....	61.0 to 62.0	363	149 000	305 000	99
Transferred from carburizing box at 1650 F. to furnace at 1250 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F.	4	.....	61.5 to 62.0	341	158 500	263 000	100
	8	.....	60.5 to 61.0	293	146 500	189 000	55
Quenched from carburizing heat. Reheated to 1450 F. Quenched and drawn at 300 F.	4	.....	62.0 to 62.5	285	177 500	290 000	138
	8	.....	62.0 to 62.5	285	139 000	269 000	72
	8	.....	63.5 to 64.0	363	235 200	268 800	79

TABLE III.—(*Continued*).

Treatment After Carburizing	Carburized at 1650 F., hr.	Case Depth, in.	Surface Hardness, Rockwell "C" Scale	Core Hardness, Brinell	Maximum Fiber Stress, lb. per sq. in.		Charpy Impact, ft-lb.
					Proportional Limit	Ultimate	
2512 STEEL							
Quenched from carburizing heat. Drawn at 300 F..................................	4	0.038	60.0 to 60.5	388	206 400	324 480	....
	8	0.054	57.0 to 58.5	363	202 000	312 500	83
	8	.....	57.0 to 60.0	388	197 000	306 500	108
Transferred from carburizing box at 1650 F. to furnace at 1350 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F..................................	4	.....	60.0	388	172 800	321 600	106
	8	.....	57.5 to 59.0	363	197 000	298 000	92
	8	.....	57.5 to 59.5	388	202 000	298 500	119
Quenched from carburizing heat. Reheated to 1380 F. Quenched and drawn at 300 F.....	4	.....	62.5 to 63.0	285	220 800	296 160	....
	8	.....	60.0 to 61.5	388	235 000	288 000	58
	8	.....	60.5 to 62.0	363	221 000	283 500	51
Cooled in carburizing box. Reheated to 1380 F. Quenched and drawn at 300 F..............	4	.....	62.0 to 63.0	311	225 600	293 760	86
	8	.....	60.5 to 61.5	388	221 000	297 000	56
	8	.....	60.5 to 62.0	363	216 000	297 000	65
Cooled in carburizing box. Reheated to 1450 F. Quenched and drawn at 300 F..............	8	.....	59.0 to 60.5	388	197 000	297 500	79
	8	.....	60.0 to 61.0	388	177 800	298 000	97
1.75 PER CENT MANGANESE-MOLYBDENUM STEEL							
Quenched from carburizing heat. Drawn at 300 F..................................	4	0.045	61.0 to 62.0	363	158 500	304 000	90
	8	0.060	59.0 to 59.5	388	182 500	269 000	61
Transferred from carburizing box at 1650 F. to furnace at 1375 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F..................................	4	.....	63.0	363	158 500	272 000	25
	8	.....	62.0 to 63.0	341	146 500	187 000	10
Quenched from carburizing heat. Reheated to 1400 F. Quenched and drawn at 300 F.....	4	.....	64.0 to 64.5	285	144 000	220 000	31
	8	.....	64.0 to 65.0	269	144 000	211 000	46
Cooled in carburizing box. Reheated to 1475 F. Quenched and drawn at 300 F..............	4	.....	64.0 to 65.0	302	158 500	221 000	18
	8	.....	64.0 to 65.0	302	115 000	206 400	18
	8	.....	63.0 to 64.0	285	158 500	197 000	17
1315 STEEL							
Quenched from carburizing heat. Drawn at 300 F.	8	0.067	58.0 to 60.5	341	144 000	286 000	16
Transferred from carburizing box at 1650 F. to furnace at 1375 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F..................................	4	0.046	62.0	331	163 000	211 000	6.5
	8	0.067	61.5 to 62.0	321	125 000	194 000	12.5
Quenched from carburizing heat. Reheated to 1400 F. Quenched and drawn at 300 F.....	8	.....	63.5 to 64.0	352	138 000	203 000	90
Cooled in carburizing box. Reheated to 1475 F. Quenched and drawn at 300 F..............	8	.....	62.5 to 63.0	341	130 000	186 000	19.5
3130 STEEL							
Quenched from carburizing heat. Drawn at 300 F.	4	0.042	62.0 to 63.0	514	211 200	211 200	14
Transferred from carburizing box at 1650 F. to furnace at 1450 F. Held until bars reached temperature of furnace. Quenched and drawn at 300 F..................................	4	.....	63.5 to 64.0	477	230 400	230 400	10
Quenched from carburizing heat. Reheated to 1400 F. Quenched and drawn at 300 F.....	4	.....	64.0 to 65.0	352	177 600	230 400	14
Quenched from carburizing heat. Reheated to 1475 F. Quenched and drawn at 300 F.....	4	.....	65.0 to 66.0	477	249 600	273 600	16

TABLE IV.—FLEXURAL STRENGTH AND ENDURANCE IN REAR AXLE TESTS COMPARED.

Test	Material	Heat Treatment	Flexural Strength, lb. per sq. in.		Number of Stress Applications Until Gear Failure in Rear Axle Test	Charpy Impact from Flexure Test Bar, ft-lb.
			Proportional Limit	Ultimate Strength		
No. 1.....	2315	Carburized 8 hr. at 1650 F. Quenched from box in oil. Reheated to 1330 F. Oil quenched. Tempered at 300 F. (Production Treatment)........	129 600	187 200	812 628	90
No. 2.....	2315	Carburized 8 hr. at 1650 F. Transferred from carburizing heat to furnace at 1375 F. Held until temperature equalized. Oil quenched. Tempered at 300 F..............................	149 000	293 000	486 802	100
No. 3.....	2315	Carburized 8 hr. at 1650 F. Quenched from box in oil. Reheated to 1475 F. Oil quenched. Tempered at 300 F..............................	182 500	295 000	403 782	99
No. 4.....	MnMo Steel	Carburized 8 hr. at 1650 F. Quenched from box in oil. Tempered at 300 F..........................	182 500	269 000	268 426	61
No. 5.....	4815	Carburized 8 hr. at 1650 F. Quenched from box in oil. Reheated to 1450 F. Oil quenched. Tempered at 300 F..............................	235 200	268 800	350 392	79
No. 6.....	4815	Carburized 8 hr. at 1650 F. Quenched from box in oil. Tempered at 300 F..........................	181 300	286 940	301 262	88
No. 7.....	1315	Carburized 8 hr. at 1650 F. Quenched from box in oil. Tempered at 300 F..........................	144 000	286 000	302 863	16
No. 8.....	2330	Carburized 4 hr. at 1650 F. Quenched from box in oil. Reheated to 1375 F. Oil quenched. Tempered at 300 F..............................	240 000	336 000	323 925	30
No. 9.....	2315	Carburized 8 hr. at 1650 F. Quenched from box in oil..	141 700	288 000	286 181	95

TABLE V.—COMPARISON OF RESULTS FROM FLEXURE TESTS AND FROM ROTATING-BEAM FATIGUE TESTS.

Curves for Plain Test Bars, Fig. 14	Steel and Treatment	Flexural Strength, lb. per sq. in.	Stress Which Results in Life of 800,000 Cycles, lb. per sq. in.	Ratio of Stress at 800,000 Cycles to Flexural Strength	Stress Which Results in Life of 10,000,000 Cycles, lb. per sq. in.	Ratio of Stress at 10,000,000 Cycles to Flexural Strength
Curve 8...	2330—carburized 6 hr. at 1650 F. Oil quenched, 1375 F. Reheat, oil quench, temper at 300 F. for ½ hr.............	322 000^a ^{a}carburized 8 hr. (0.050 in.)	180 000	0.558	157 000 0.041-in. case	0.487
Curve 9...	2330—carburized 3 hr. at 1650 F. Oil quenched, 1375 F. Reheat, oil quench, temper at 300 F. for ½ hr.............	336 000^a ^{a}carburized 4 hr. (0.035 in.)	177 000	0.527	147 000 0.030-in. case	0.438
Curve 4...	2315—carburized 6 hr. at 1650 F. Oil quenched, 1475 F. Reheat, oil quench, tempered at 300 F. for ½ hr...........	295 000^a ^{a}carburized 8 hr. (0.053 in.)	137 000	0.465	120 000 0.041-in. case	0.407
Curve 5...	2315—carburized 6 hr. at 1650 F. Cool in box, 1475 F. Reheat, oil quench, temper at 300 F. for ½ hr.............	296 000^a ^{a}carburized 8 hr. (0.053 in.)	133 000	0.448	115 000 0.041-in. case	0.388
Curve 7...	2330—carburized 3 hr. at 1650 F. Transferred to furnace at 1375 F. Oil quench, tempered at 300 F. for ½ hr...........	266 000^a ^{a}carburized 4 hr. (0.035 in.)	130 000	0.488	113 000 0.031-in. case	0.425
Curve 1...	2315—carburized 6 hr. at 1650 F. Oil quenched from box, tempered at 300 F. for ½ hr...........................	288 000^a ^{a}carburized 8 hr. (0.053 in.)	130 000	0.452	108 000 0.041-in. case	0.375
Curve 2...	2315—carburized 6 hr. at 1650 F. Transferred to furnace at 1375 F. Oil quench, transferred at 300 F. for ½ hr..........	293 000^a ^{a}carburized 8 hr. (0.053 in.)	127 000	0.433	105 000 0.041-in. case	0.358
Curve 3...	2315—carburized 6 hr. at 1650 F. Oil quenched, 1330 F. Reheat, oil quench, temper at 300 F. for ½ hr.............	187 200^a ^{a}carburized 8 hr. (0.053 in.)	117 000	0.625	101 000 0.042-in. case	0.540
Curve 3A .	2315—carburized 3 hr. at 1650 F. Oil quenched, 1330 F. Reheat, oil quench, temper at 300 F. for ½ hr.............	197 700^a ^{a}carburized 4 hr. (0.041 in.)	105 000	0.531	98 500 0.032-in. case	0.498

be made with a similarly stressed test specimen. The difference between the calculated gear tooth stress and the stress in a test specimen necessary for failure in 800,000 cycles when stressed in tension only would be even greater than shown by the rotating-beam fatigue test.

If we assume for the purpose of making a comparison that the actual stress developed in the gear tooth under low gear torque was 117,000 lb. per sq. in., then the life to be expected from other materials whose fatigue test curve is shown in Fig. 15 would be as shown in Table VI. The actual life obtained is also given for some of these materials.

On the basis of stress-life results obtained in rotating-beam fatigue tests, the life obtained in rear axle tests indicates that the gear teeth in all of the experimental gears were stressed much higher than in the standard gear which is listed at the bottom of Table VI. This deduced stress mounted to 189,000 lb. per sq. in. in the gear made of 2330 steel, oil-quenched from the carburizing heat, reheated to 1375 F., oil-quenched and drawn at 300 F. In passing it is interesting to note that the steel and treatment just mentioned gives a life of more than 10,000,000 stress cycles of 150,000 lb. per sq. in., exceeding by a considerable margin the figure of 110,000 lb. per sq. in. given by von Heydekampf[1] as an endurance limit seldom exceeded.

Table VI.—Life Indicated by Rotating-Beam Fatigue Tests Compared to Actual Life Obtained in Gear Tests.

Curves for Plain Test Bars, Fig. 14	Stress Applications Expected at 117,000 lb. per sq. in.	Stress Applications, Actual Life in Rear Axle Test	Stress on Rotating-Beam Specimen Necessary for Failure in Number of Cycles, Life on Rear Axle Test, lb. per sq. in.
Curve 8	over 100 000 000	......	......
Curve 9	over 100 000 000	323 925	189 000
Curve 4	16 000 000 or more	403 782	137 000
Curve 5	7 000 000	......	......
Curve 7	6 000 000	......	......
Curve 1	3 500 000	286 181	140 000
Curve 2	2 200 000	486 802	127 000
Curve 3	800 000	812 628	117 000

Apparently the plain rotating-beam fatigue test is of no use in selecting materials for rear axle gears.

Tests on notched rotating-beam fatigue specimens were then conducted to test the second hypothesis that tooth shape and tool marks caused more stress concentration in some steels than in others. Before making many tests it appeared desirable first to determine the type of notched specimen to use.

Comparison of the calculated stress in the gear and the life obtained in rear axle test with the stress necessary to obtain the same life in a plain rotating-beam fatigue test indicates a stress concentration of about four times. It was desired therefore to obtain a design of notched test bar which would produce a stress concentration of that amount and which would produce a slope of the slanting part of the *S-N* curve parallel to the curve obtained by plotting the results of rear axle tests.

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[1] G. S. von Heydekampf, "Properties of Alloys Under Dynamic Stresses," Conference on Metals and Alloys. Case School of Applied Science, Cleveland, Ohio, *Paper No. 16*, November, 1931.

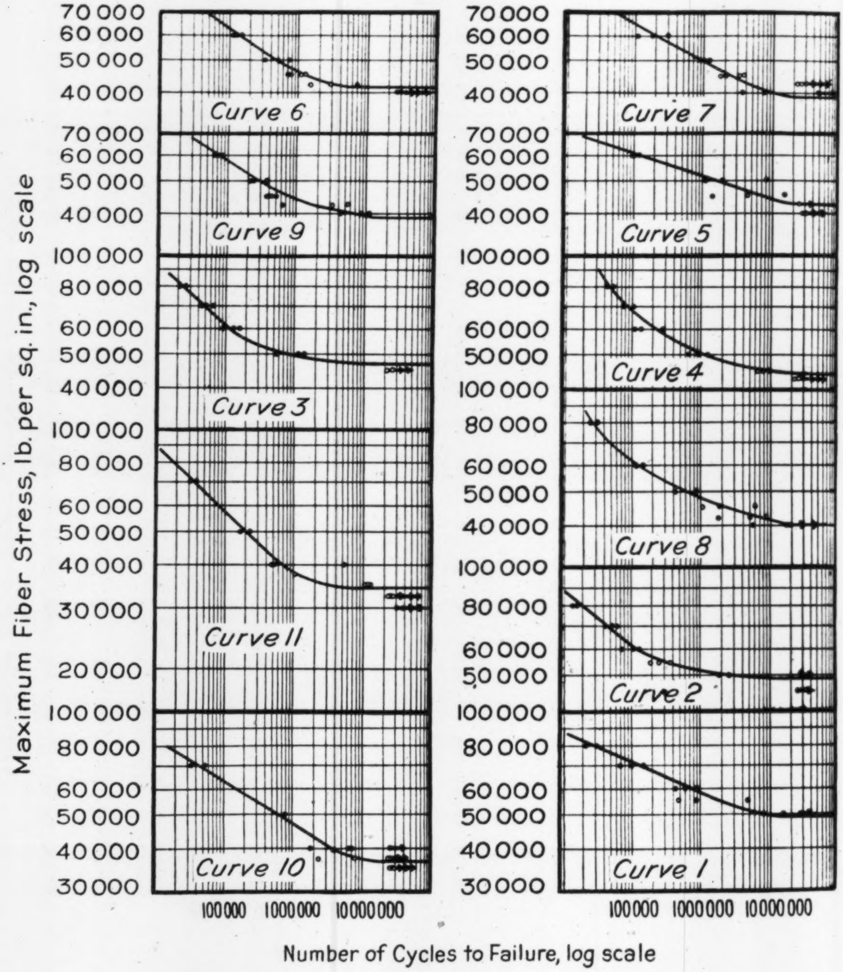

FIG. 16.—Rotating-Beam Tests of Notched Bars.

Four specimen designs (shown in Fig. 22) were tested using two steels and heat treatments. Figure 17 shows how these different notches produced stress concentration. Specimen design No. 4 was selected as being the nearest approach to the type of curve obtained from gear tests.

Eleven combinations of steel and heat treatments were investigated with this type of specimen with the results summarized in Fig. 14. Plots of individual points for each curve are shows in Fig. 16.

In order to use the same method of comparison as was used when comparing plain rotating-beam fatigue tests with gear tests, we will select the stress which causes failure in 800,000 cycles for the 2315 steel test bars treated like the regular production gears.

The stress-cycle relation for this steel and treatment are shown in curve 9, Fig. 14, where a calculated stress of 45,000 lb. per sq. in. causes failure in 800,000 cycles. The cycles to failure for other steels and treatments at a stress of 45,000 lb. per sq. in. are given in Table VII. For comparison, are also given the actual gear test results obtained in rear axle life tests as well as the calculated stress taken from the notched-bar endurance curves corresponding to the life obtained in the gear tests.

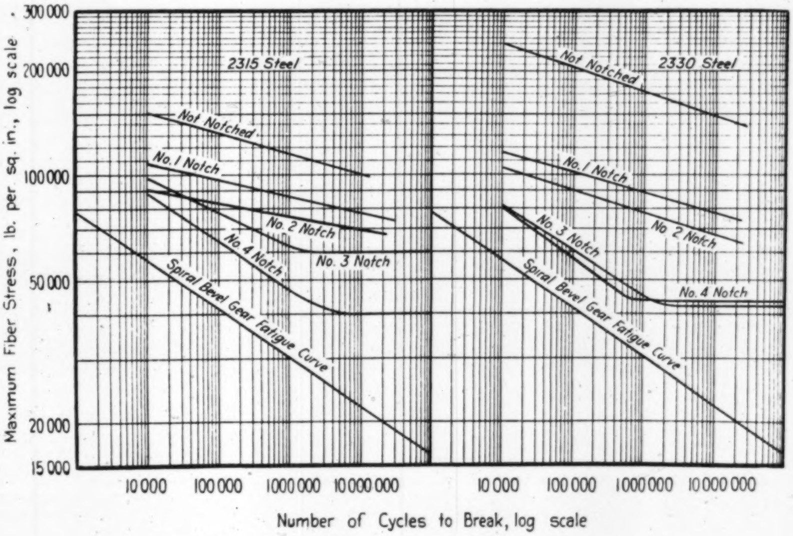

FIG. 17.—Effect of Test Bar Design on Stress Concentration.

2315 Steel—Carburized 8 hr. at 1650 F.
Oil quenched from carburizing heat.
Reheated to 1330 F.
Oil quenched—300 F. draw.

2330 Steel—Carburized 4 hr. at 1650 F.
Oil quenched from carburizing heat.
Reheated in furnace to 1375 F.
Oil quenched—300 F. draw.

It is interesting to note in comparing the results of notched and plain rotating-beam fatigue tests that the notch has a more damaging effect on strong steels with hard cores than it has on weaker ones with soft cores, as illustrated in Table VIII.

Moore and Henwood,[1] Kommers,[2] Lessells,[3] Krouse,[4] Lehr,[5] and McAdam[6] and others observed that the same notch caused different degrees

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[1] H. F. Moore and P. E. Henwood, "The Strength of Screw Threads Under Repeated Tension," *Bulletin No. 264*, Engineering Experiment Station, University of Illinois, Urbana, Ill., March 13, 1934.

[2] J. B. Kommers, "The Effect of Under-Stressing on Cast Iron and Open-Hearth Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 368 (1930).

[3] Discussion by J. M. Lessells of paper by R. R. Moore, "Resistance of Metals to Repeated Static and Impact Stresses," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 606 (1924).

[4] G. N. Krouse, "A High-Speed Fatigue Testing Machine and Some Tests of Speed Effect on Endurance Limit," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 156 (1935).

[5] Ernst Lehr, "Surface Sensitiveness and Internal Absorption of Energy During Endurance Test," *Zeitschrift für Metallkunde*, February, 1928.

[6] D. J. McAdam, Jr., "Fatigue and Corrosion-Fatigue of Spring Material," *Transactions*, Am. Soc. Mechanical Engrs., Applied Mechanics Division, Vol. 51, Part I, January–April, 1929, p. 45.

TABLE VII.—LIFE INDICATED BY NOTCHED ROTATING-BEAM FATIGUE TESTS COMPARED TO ACTUAL LIFE OBTAINED.

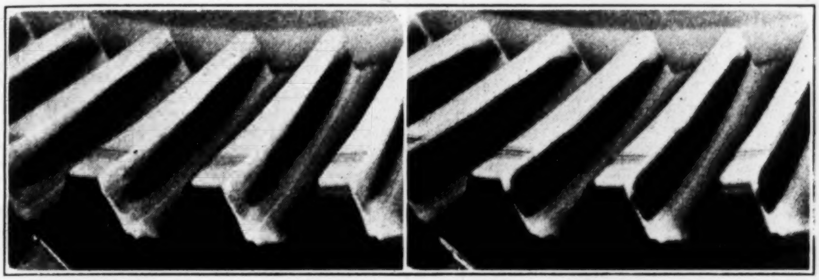
Curve for Notched Test Bars, Fig. 14	Steel and Treatment	Number of Stress Applications Expected at Stress of 45,000 lb. per sq. in.	Number of Stress Applications to Failure in Rear Axle Gear Test	Stress Required for Failure of Notched Test Bar in Number of Cycles Given in Previous Column
Curve 9.....	Regular gear steel and treatment. 2315—carburized 8 hr. at 1650 F., oil quenched, reheated to 1330 F., oil quenched, 300 F. temper....................	800 000	812 628	45 000
Curve 10....	1315—carburized 8 hr. at 1650 F., oil quenched, 300 F. temper..................................	1 500 000	302 863	55 000
Curve 11....	1.75 per cent manganese-molybdenum steel, carburized 8 hr. at 1650 F., oil quenched, 300 F. temper......	350 000	268 426	47 000
Curve 6.....	2315—carburized 8 hr. at 1650 F., oil quenched, 300 F. temper..................................	1 300 000	286 181	55 000
Curve 8.....	3115—carburized 8 hr. at 1650 F., oil quenched, 300 F. temper..................................	2 200 000	......	......
Curve 7.....	2315—carburized 8 hr. at 1650 F. Transferred from carburizing heat to furnace at 1375 F., oil quenched, 300 F. temper................................	2 900 000	486 802	55 000
Curve 4.....	2512—carburized 8 hr. at 1650 F., oil quenched, 300 F. temper..................................	5 000 000	......	......
Curve 5.....	2330—carburized 4 hr. at 1650 F., oil quenched, 1375 F. reheat, oil quenched, 300 F. temper..............	7 000 000	323 925	55 500
Curve 3.....	3115—carburized 8 hr. at 1650 F., oil quenched, reheat to 1475 F., oil quenched, 300 F. temper...........	over 10 000 000	......	......
Curve 2.....	4620—carburized 8 hr. at 1675 F. Transferred to cyanide at 1400 F., oil quenched..................	over 10 000 000	......	......
Curve 1.....	4620—carburized 8 hr. at 1675 F., oil quenched......	over 10 000 000	......	......

TABLE VIII.—STRESS CONCENTRATION CAUSED BY NOTCHED BARS.

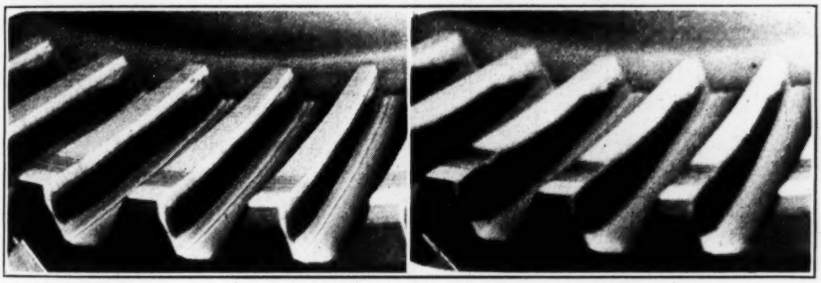
Steel and Heat Treatment	Stress at which Failure Occurs in 800,000 Applications, lb. per sq. in.		
	Plain Test Bars	Notched Test Bars	Stress Concentration
2330—carburized 4 hr. at 1650 F.; oil quenched, 1375 F. Reheat—oil quenched—300 F., draw..................................	177 000	52 000	3.4
2315—carburized 8 hr. at 1650 F.; oil quenched, 1330 F. Reheat—oil quenched—300 F., draw..................................	117 000	45 000	2.6
2315—carburized 8 hr. at 1650 F. Transfer to furnace at 1375 F., oil quenched—300 F. draw..................................	127 000	51 500	2.47
2315—carburized 8 hr. at 1650 F., oil quenched—300 F. draw..............................	130 000	47 000	2.77

of stress concentration in different metals, usually causing greater concentration in stronger, harder metals. That this is not always so is pointed out by Moore and Lyon.[1] Copper for example has high ductility and is greatly damaged by notches.

The second hypothesis that notches cause more stress concentration in some steels than in others has received supporting evidence in the tests described, but notched rotating-beam tests do not enable us to predict the gear life in a rear axle test although they do serve as a much better guide for selecting gear steels than the plain rotating-beam test. This is indicated in Fig. 14 where the results of notched-bar tests show much closer grouping than the results of transverse or plain fatigue specimens.



(*a*) Tooth Contact at Light Load Gear A. (*b*) Tooth Contact at Heavy Load Gear A.



(*c*) Tooth Contact at Light Load Gear B. (*d*) Tooth Contact at Heavy Load Gear B.

FIG. 18.—Gear A Shows Better Tooth Contact than Gear B.

Referring to Table VII it will be observed that although the discrepancy between actual life in gear tests and life indicated by notched-bar fatigue tests is large, the difference in amount of stress required to cause this variation in life is not so large. The small increase in stress which accounts for this difference could well be accounted for by the third hypothesis which presumes that distortion in heat treatment and excessive deflection under load causes additional stress increment.

It seems reasonable that the difference in indicated gear stress shown in the last column in Table VII is due almost entirely to variations in tooth bearing as the result of distortion in heat treatment because all these gears were generated with the same cutting tools and supported alike in the same rear axle assembly.

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[1] H. F. Moore and S. W. Lyon, "Tests of the Endurance of Gray Cast Iron Under Repeated Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 87 (1927).

The way in which tooth contact varies is shown in Fig. 18. Fig. 18 (*a*) and (*b*) show a better contact at light and full load respectively, characterized by light contact at light loads at the heavy end of the tooth, and uniform contact over the whole length of the tooth at full load. Figure 18 (*c*) and (*d*) show poorer contact at light and full loads characterized by excessive contact at the heavy end of the tooth at light load and concentration of stress at the heavy end of the tooth at full load. When tooth contacts are like those in Fig. 18 (*a*) and (*b*), longer life is usually obtained than when contacts are like those shown in Fig. 18 (*c*) and (*d*).

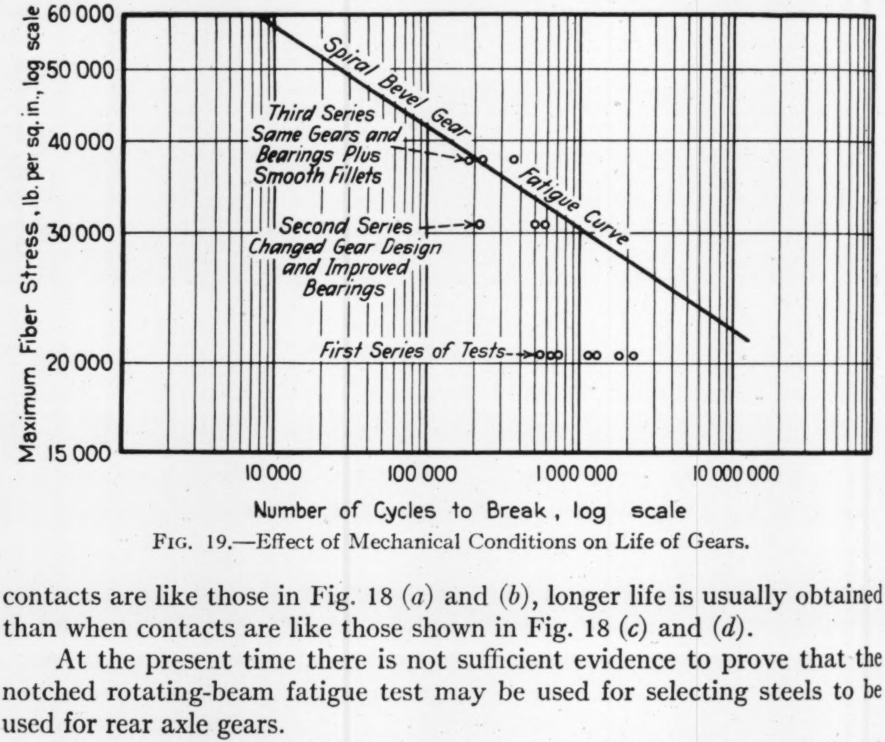


FIG. 19.—Effect of Mechanical Conditions on Life of Gears.

At the present time there is not sufficient evidence to prove that the notched rotating-beam fatigue test may be used for selecting steels to be used for rear axle gears.

The lack of agreement between gear test results shown in Table I and rotating notched-bar fatigue test results shown in Fig. 14 is presumably due to variation in stress concentration as a result of different condition at the base of teeth and to variation in point of load application on the gear tooth due to distortion and to difference in total deflection from one rear axle design to another. If these variations could be reduced it is probable that a notched-bar fatigue test would be in closer agreement with rear axle tests.

The sensible thing to do is to pick a steel with due regard to the possibility of heat treating that steel without undue distortion. Even though a steel that possesses satisfactory strength gives a short life when tested in a rear axle gear for the first time, it will pay to experiment with the design and position of bearings supporting the pinion and ring gear, with method of generating teeth, and heat treatment methods, long enough to work out the conditions necessary to give the desired tooth contact. When this has been worked out the life in the rear axle will be satisfactory. The experience with rear axle gears in one car illustrates this point. Steel G.M.C 4615-A was adopted for the ring gear and drive pinion so designed that the calculated stress in the pinion was 20,800 lb. per sq. in., which according to the fatigue curve for spiral bevel gears should have resulted in a life of about 17,000,000 cycles, but due to unsatisfactory support of these gears, the stresses in the pinion were increased so that the average of 7 tests was a life of 1,086,000 (see Fig. 19). The gears should have lasted that long when stressed to 30,000 lb. per sq. in. Therefore, it might be said that they were 30 per cent poorer from a stress standpoint than the average for all gears tested in spite of the fact that 4615 steel possesses of itself a high resistance to repeated concentrated loads, as determined by laboratory tests. A redesign of the bearings and the gear, still using the same material and heat treatment, resulted in a life of 440,000 stress applications when stressed at 30,200 lb. per sq. in. calculated. Referring again to the average curve, shown in Fig. 19, these gears should have lasted that long for a stress of 34,000 lb. per sq. in., so these gears were only 11 per cent poorer than the average. In other words an improvement of 19 per cent had been made. Then by shaping the tooth generating tools so that the depth of sharp tool marks at the base of the teeth was greatly reduced, the gears then lasted 295,000 stress applications of 36,800 lb. per sq. in. This was better than the average curve which indicates a similar life when stressed at 36,000 lb. per sq. in., or 2.2 per cent better than the average. This represents another 13.2 per cent improvement. Other mechanical modifications could probably be made to increase the life of this pinion still further until conditions were such that stress concentration is reduced as low as possible. Until the influence of mechanical factors is reduced to the same magnitude as the difference between steels, we shall be unable to improve the life of the gear by changing the steel and heat treatment.

Work has been done also on developing the proper cutting and heat treatment for 3115 steel to obtain life equal to that indicated by the average curve for spiral bevel gears. The work thus far has shown that when proper tooth contacts and fillets are obtained this material's durability is equal to the general average of other gear steels tested.

APPENDIX

Preparation and Testing of Flexure Test Specimens:

The bars were first finished to 0.5000 by 0.625 by 5 in. and copper plated over all after which the 0.625-in. dimension was reduced to 0.5000 in. by removing equal amounts of material from the two sides. Finishing to 0.5000 in. in each instance was accomplished by grinding opposite sides. Removal of the copper from two opposite sides in this manner resulted in carburization of only two sides after which the samples were subjected to various heat treatments.

The test consisted of applying a load to one of these carburized and hardened sides at a point midway between two supports placed 4 in. apart. The deflection for each 400-lb. increase in load was determined up to 2000-lb. load and for every 200-lb. increase from 2000 to 3000-lb. load. Above 3000-lb. load, the deflection was determined for each 100-lb. increase until the results indicated that the applied load was well beyond the proportional limit. The proportional limit load was readily determined from a load-deflection curve.

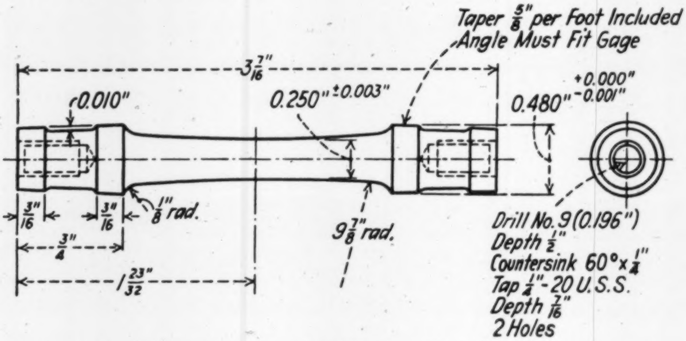


FIG. 20.—Standard Fatigue Specimen.

Surface hardness as measured by both a file and Rockwell hardness test was also determined on each bar together with the Brinell hardness of the core.

Oil of 60 to 80 sec. viscosity at 100 F. was used at a temperature of 90 to 100 F. as a quenching medium for all specimens.

The tests for the most part were not run in duplicate. Where duplicate samples were run, excellent agreement was obtained in ultimate strength, but in some cases appreciable variation occurred in proportional limit values. Ultimate strength being the property indicative of resistance to fatigue, lack of agreement in proportional limit determinations was not considered important. Since the flexure tests were merely a means of indicating trends it was considered unnecessary to run check tests.

Impact Test:

Specimens without notches for the Charpy impact test were made from the halves of the broken flexure test bars. The specimens were ground to 0.394 in. thick and made 2.10 in. long. The hardened case was left on one side and all ground off the other side. The side without the case was used for the striking side.

Rear Axle Gear Endurance Tests:

Three drive pinions of each material and heat treatment were assembled in rear axle third members all of which were passed as acceptable by the regular production inspection with respect to tooth bearing on the drive side before acceptance for test purposes. It was noted by the inspector that the tooth contacts were not quite as good as the regular production gears.

These third member assemblies were then tested on the rear axle dynamometer at a propeller shaft speed of 220 r.p.m. and a propeller shaft torque of 405 lb-ft., equivalent to a calculated pinion tooth stress of 29,130 lb. per sq. in. A-150 oil was used as a lubricant and the differential carriers were cooled by two fans in such manner that the temperature of the lubricant did not exceed 165 F.

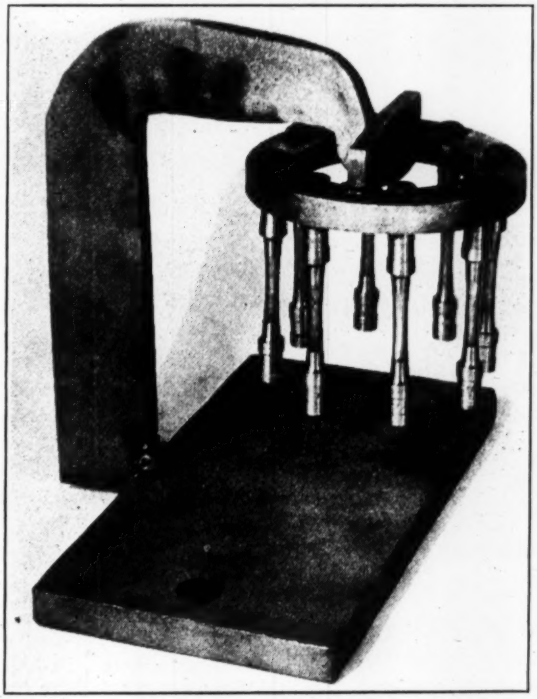


FIG. 21.—Fixture for Supporting Test Specimens During Heat Treatment.

Contact studies were made of each drive pinion tested, under the following light and medium loads before testing under the heavy load conditions just described. These contact studies are made as a regular part of our rear axle test procedure.

(*a*) Light load—66 lb-ft., propeller shaft torque at 112 r.p.m.

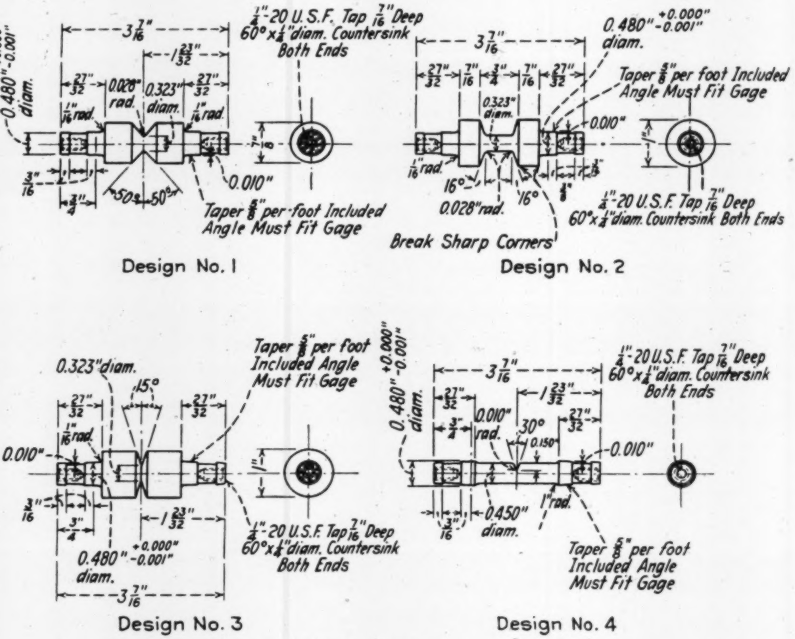
(*b*) Medium load—203 lb-ft., propeller shaft torque at 162 r.p.m.

Rotating-Beam Fatigue Tests:

Fatigue tests were made on R. R. Moore machines. Although the design of the standard specimen was modified for these tests by changing the diameter at the midpoint from 0.300 in. to 0.250 in., the specimens are considered as being of standard design to differentiate between them and specimens of the notched design which was later adopted for these tests. The change to a smaller diameter was necessary since the capacity of the test machines did not permit enough loading of carburized and hardened 0.300-in. diameter specimens to establish an *S-N* curve between 100,000 and 10,000,000 reversals. The standard specimen is shown in detail in Fig. 20.

It was decided to use the same carburizing compound for test specimens as is used in production for carburizing drive pinions. Thus, the case of the test specimens, at least with respect to the carbon concentration at the surface, was comparable to the case of production pinions. Production practice is to carburize drive pinions to a depth of 0.050 to 0.055 in. This case depth, however, was not advisable on the 0.250-in. diameter specimens since the ratio of case to core would be so much greater than the ratio of case to core at the base of drive pinion teeth. The specimens were, therefore, carburized to a depth of 0.040 to 0.044 in., which was obtained in 6 hr. at 1650 F.

The test specimens were carburized and heat treated in lots of eight. Before carburizing, all except the 2512 steel specimens were semi-finished by a polishing operation in which circumferential tool marks were almost completely eliminated. Those specimens, which were subsequently reheated in a furnace, were finish polished after final heat treatment and tool marks completely removed as judged with a binocular microscope.



0.480" +0.000" -0.001" diam.
3 7/16"
1 23/32"
27/32"
0.028" rad.
0.323" diam.
1/16" rad.
1/4"-20 U.S.F. Tap 7/16" Deep 60°x1/4" diam. Countersink Both Ends
Taper 5/8" per foot Included Angle Must Fit Gage
0.010"
3/16"
3/4"
50°
60°
7/16"
16°
0.028" rad.
Break Sharp Corners
15°
30°
0.150"
0.450" diam.
1" rad.

Design No. 1

Design No. 2

Design No. 3

Design No. 4

FIG. 22.—Notched Specimen Designs.

The 2315 steel specimens that were reheated in cyanide were finished after quenching from the carburizing heat and tested without additional polishing. It was necessary to finish the 2512 steel specimens completely before carburizing, since they were transferred to cyanide which made it inadvisable to do anything to the surface after heat treatment.

A special fixture was designed to which eight test specimens could be attached during both carburizing and hardening. During carburizing, this fixture was supported by lugs on the inside of a cylindrical container slightly larger in diameter than the outside of the fixture. During reheating in a furnace, the fixture was hung on a special stand as shown in Fig. 21. The fixture was also used during heating in cyanide, suspended from a rod on top of the cyanide pot. The length of the carburizing cycle was in each instance one hour longer than the time at heat and the length of time in the reheating or transfer operation was maintained the same for each group of specimens similarly treated. Those specimens which were heated in or transferred to cyanide were in the bath 10 min. Heat treatments were, therefore, conducted to minimize metallurgical variations in the various lots given a specific treatment and to minimize as well as maintain distortion as uniform as possible.

After heat treatment the centers at the ends of all specimens were lapped so that the test length ran true within 0.001 in. on the test machines. The shoulders of the specimens were then ground as indicated in Fig. 20.

Notched Rotating-Beam Fatigue Tests:

The four types of notched fatigue test bars tested to determine which one should be selected for further testing are shown in Fig. 22.

All machining was done before heat treating because this is the practice followed in making spiral bevel gears.

Stresses used for plotting *S-N* curves for notched specimens, design Nos. 1, 2 and 3, Fig. 22, were obtained by using the same beam formula as was used for the plain test bar, using the thickness at the bottom of the notch as the diameter of the test bar. Stresses in specimen design No. 4, which was adopted for subsequent testing, were calculated at the surface opposite the notch. Because of the shape of the cross-section through the notch, this point is more highly stressed than in the center of the notch. Failure starts however at the intersection of the bottom of the notch with the surface where the stress is apparently highest. No method is known for calculating stress at that point; therefore the value obtained for the surface opposite the notch was used in plotting curves.

DISCUSSION

Mr. R. E. Peterson[1] (*presented in written form*).—The proper judgment of engineering problems is really an art. The accumulation and analysis of data are not as important as the initial judgment of the problem and the resulting decisions as to what to do and what not to do. For example, in considering the rear axle gear problem, it would be just as easy to accumulate and analyze the same "man-hour amount" of data on impact as on fatigue. The difference is that as far as the problem at hand is concerned the fatigue program properly carried out leads to a solution, whereas the impact program would be meaningless, or worse in that it could give misleading results. The initial judgment of this problem based on a study of service records and on a study of the dynamics of the system is worthy of careful study by engineers.

At the end of the paper, the statement is made that no method is known of calculating the stress in the notch of the specimen denoted as "design No. 4" (page 135). An approximation can be obtained by means of the Neuber[2] solution, which gives a stress concentration factor of 3.14 for the two-dimensional case and 3.47 for the three-dimensional case. It is interesting to note that the average fatigue stress concentration factor obtained by dividing the endurance limit (10,000,000 cycles—plain test bars—Fig. 14) by the notch endurance limit (Fig. 14) is about 3.

In a paper before the June, 1935, meeting of the American Society of Mechanical Engineers in Ann Arbor, the writer presented results which showed that in many cases theoretical stress concentration factors were realized in fatigue tests. One or more of three conditions were fulfilled in these cases: large specimens, alloyed or heat-treated materials. An idea was put forth that the criterion was perhaps the number of grains in the region of peak stress. Since orientation of grains is involved in the mechanism of failure, a relatively small number of grains at peak stress may result in a higher endurance strength. It is interesting to note that in Table I of the paper, the highest value was obtained with coarse-grained material. It is understood that some other variables also entered into the picture so that a definite conclusion cannot be drawn. However, it is clear that the effect of grain size should be investigated thoroughly in further work on the stress concentration problem in fatigue.

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[1] Manager, Mechanics Division, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] H. Neuber, "Der Räumlich Spannungszustand in Umdrehungskerben," Ingenieur Archiv., Vol. 6, p. 133 (1935).

MR. W. H. SWANGER[1] (*presented in written form*).—The data in this paper are exceptionally interesting and valuable in showing how far the endurance properties actually realized in a very common type of machine member fall short of the endurance strength it is possible to attain in the materials available for such parts. The paper will be a comfort to any metallurgist who has had difficulty in convincing engineers that failures of certain types were not the result of defective material. The following points, which were well brought out in the paper, appear to be of particular importance in the general problem of failures in metals: (1) in any study to provide means for overcoming or avoiding a prevalent type of failure, the collection and intelligent interpretation of service data is of first importance; (2) because the effect of repeated applications of maximum stress is cumulative, the service life is determined by the number of cycles of the maximum stress, whether applied intermittently or continuously, and fatigue failures in steel machine members may occur after many more than 10,000,000 cycles of operation if there have been occasional periods when the stresses exceeded the average for normal service; (3) a knowledge of the behavior of a steel under repeated stresses considerably above the fatigue limit may be of more practical use to the designer than the numerical value of the fatigue limit; (4) it is of great importance to study a design critically to see if by any modification of general shape, or more care in finishing—avoiding hogging cuts and rough surfaces—dangerous stress concentrations can be reduced.

In Part II of the paper, pages 127 to 129, the observation is made that strong hard metals have, in general, greater notch-sensitivity than softer more ductile metals. Soft copper with high ductility has notoriously high notch sensitivity and is cited as an exception. In a recent paper,[2] D. J. McAdam, Jr., pointed out that ductility in itself is not a measure of the degree of notch sensitivity of a metal but that a derived quantity, named by him "percentage strength" and which is a measure of tensile work-hardening capacity, does appear to be closely related to notch sensitivity. He also showed why soft copper or other fully annealed metals and single-phase alloys need not be considered real exceptions to his hypothesis that notch sensitivity is closely related to percentage strength. Possibly if data were available on the tensile work-hardening capacity (percentage strength) of the steels investigated by the authors, there would be a rational explanation, on this basis, for the differences in behavior of the steels in the axle gear tests and the results of the notched-bar fatigue tests.

MR. T. MCL. JASPER.[3]—This paper gives some idea of the corelation between fatigue tests using service samples and fatigue tests made on simple samples of given materials, using the rotating-beam or cantilever machines. The first method of testing using service condition may be assumed to give the answer as to the quality of materials, heat treatments and designs, and is, by every means, the most satisfactory method for obtaining the correct answer. The second method of testing using a rotating-beam fatigue machine can be considered to select only materials of high fatigue quality. The other desirable qualities for the service, such as, toughness, wear resistance, and resistance to repeated shock, cannot be obtained in a simple fatigue machine. These other properties may be just as important as the fatigue values of the material.

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[1] Metallurgist, National Bureau of Standards, Washington, D. C.

[2] D. J. McAdam, Jr., and R. W. Clyne, "Influence of Chemically and Mechanically Formed Notcnes on Fatigue of Metals," National Bureau of Standards *Journal of Research*, Vol. 13, October, 1934, Research Paper 725.

[3] Director of Research, A. O. Smith Corp., Milwaukee, Wis.

It is appropriate to draw attention to the fact that where designs, such as gear teeth are involved, reentrant angles will have a more serious effect in reducing the life of certain materials than it will when using other materials. Materials that are "notch sensitive" although they may give high fatigue values in a simple fatigue test when compared to other materials, may also give considerably lower values under a service type of test, such as the authors have resorted to. A notched fatigue specimen cannot give as good an answer to the designed service part.

I was very much impressed by the authors' statement in regard to grain size. I am quite in agreement with the fact brought out by the authors that one cannot use grain size as a criterion of value in fatigue. The question of finish which every investigator of fatigue has recognized is of course brought out by the tests and should be recognized very much more than has generally been done. Referring to the Charpy notched-bar impact test, the practical application of such a test should give it less value than many metallurgists and engineers seem to do. I want to emphasize this point because I am very much of the opinion that the Charpy notched-bar impact test has very little engineering value.

MR. L. B. TUCKERMAN.[1]—This paper is one which should be studied carefully, and most carefully by every man who has to deal with moving parts which may be subject to fluctuating stresses. There are two conclusions which I wish to point out even in more detail: We examine all sorts of fatigue failures at the National Bureau of Standards, and are supposed to try to find out what caused them. We analyze the possible cycles of stress and in many cases we find that the various cycles of stress which could come upon the structure give values of perhaps a hundred million or two hundred million or three hundred million cycles or something like that. We know, however, that if a steel can endure a given stress fluctuation for ten million cycles of stress, it should endure the same fluctuation forever or at least should not fail in any reasonable finite time. That has been verified by many experiments. On that basis we cannot understand why these structures failed. Then we analyze more carefully and find that the conditions of fluctuating stress are not uniform, that there are times in which because of resonance the structure is subject to stresses enormously higher than at other times. Although the total number of cycles of stress may be several hundred million, the number of cycles in which resonance produces these enormously higher stresses is many times less. Putting it on that basis, we are able to understand why the failure comes after a long life.

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[1] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

One other thing about this type of failure is that in examining what are supposedly identical structures subject to identical service you find one of them failing after perhaps one hundred hours of service and another one failing after perhaps two thousand hours of service, the difference being that one has happened to come into conditions of high stress much more frequently than the other. In these cases there is no definite relation between length of actual service and the occurrence of fatigue failure.

The other conclusion is: until the influence of the mechanical factors is reduced to the same order of magnitude as the difference between steels, we shall be unable to improve materially the life of the gear by changing the steel and heat treatment. We have at the Bureau of Standards what is called our chamber of horrors; case after case where engineers, who ought to know better, designed structures with sharp re-entrant angles, with rough tool marks at the bottom of fillets, with sharp outstanding fins and rough corners on those fins, and still they want to know why metallurgists do not give them better materials. Until they have learned their lesson, what is the use of making better materials?

MR. W. P. EDDY, JR.[1] (*presented in written form*).—It may be difficult for many of us who are concerned with materials to accept statements, even so well supported as are those of the authors, to the effect that the life of axle gears cannot be improved by changing steels and heat treatments alone. Almost every gear engineer and metallurgist can recall so many instances where fatigue failures in service have been decreased by merely changing pinion steels or heat treatments, that it is not quite satisfying to assume that accidental changes in mechanical conditions which may have occurred simultaneously with the metallurgical changes had invariably beneficial results. This paper indicates that closer observation of all conditions in the future may be advisable in order to determine the true cause of any improvement in gear performance.

I understand from the text of the paper that imperfect mechanical conditions, such as elastic deflection of axle parts, surface scratches, eccentric assemblies, etc., are considered as having pronounced effects on gear life. How is it possible, then, that conditions which affect performance so markedly and which cannot be the same in each of 62 ordinary production axles of many sizes and designs permit stresses, calculated from fatigue test results on those axles by the modified Lewis or any single formula, to fall so close to an ideal fatigue endurance curve?

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[1] Metallurgist, General Motors Truck Co., Pontiac, Mich.

The indication that S.A.E. No. 3115 steel can be used, at lower cost, to produce gears equal to those of higher alloy steels, leads to the thought: Why stop at No. 3115? Is it believed possible to develop a 1½ per cent manganese or a 1 per cent chromium case-hardening steel, by decreasing the variables in steel making and in heat treating in order to reduce and control distortion, to the extent that gears of these low-priced steels equal to those made of accepted axle gear steels can be produced? This paper may be pointing the way to manufacturing economies hitherto believed impossible of attainment.

MR. H. W. MCQUAID[1] (*presented in written form*).—This paper indicates clearly that the old idea that rear axle gears require steels of a maximum toughness and ability to absorb shocks was erroneous. It is a great pleasure to be able to state that the results which Messrs. Almen and Boegehold show have been checked many times in service, not only in passenger car applications, but also in truck and bus application. One who has carefully studied the many failures which occur in bus and truck rear axle gears, soon realizes that most of the failures are accompanied by highly localized pressures on the contacting surfaces of the teeth which either cause rapid wear and pitting or else greatly increased stresses at the root of the tooth.

The photographs shown in Fig. 11 of the paper indicate what takes place when deflection occurs. The contact pressure under heavy loads moves to the top of the heel end of the tooth, so that not only is the stress greatly increased at the root of the tooth, but high slide and very high localized pressures occur at the points of initial contact. If this movement is severe enough, we generally find either rapid fatiguing of the teeth at the root, which may or may not be accompanied by heavy wear, or else heavy pitting may occur at the points of maximum surface pressure.

It is quite obvious if the supporting members are not so designed that they maintain proper tooth contact under heavy loads, that the life of the gears will be much shorter than necessary, and problems of scoring, noise, etc., will be considerably increased.

In inspecting gears which have failed, it becomes very obvious that the great majority of failures are not due to metallurgical difficulties, but are due primarily to either improper assembly or deflection in service. It is obvious of course that gears which are too soft, and hence wear rapidly, soon approach a condition which corresponds to that of extreme deflection, in that pressures tend to become localized so that wear increases at a very rapid rate and fatigue occurs in a much shorter time than usual. Close study of hundreds of rear axle gear failures indicates that more can be done to improve the performance of spiral bevel gears by increasing the rigidity of mounting and by proper selection of spiral and pressure angles, etc., than by any metallurgical changes of which we now have knowledge.

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[1] **Metallurgist, Republic Steel Corp., Massillon, Ohio.**

Those tests which were formerly given gear steels as a basis of selection and which involve impact tests and core property tests were, for passenger car service at least, not only highly misleading but resulted in high rejections of steel which would undoubtedly have been satisfactory and also in the unnecessary expenditure of large sums of money for large alloy additions which were of little value.

In the truck and bus field, particularly where the design was such that the stresses exceeded those indicated as being satisfactory, as shown in Fig. 17, many failures occurred which were of a somewhat different type than those indicated in this paper. Thus it was found that with one type of truck rear axle gear used in extremely large quantities, the life of the gear could be considerably increased by metallurgical means. It is true that a change in design would permit the use of simpler steels and simpler heat treatment, but with the design as it was, it was found that the life of the gears could be increased as much as 300 per cent by metallurgical means.

It has been found in truck applications, particularly where under certain conditions full load is applied in low gear for relatively long periods of time, that the temperature developed on the surface of the pinion tooth is frequently high enough to cause a softening on the surface with resultant increased wear. On the truck ratios, the pinion is lubricated generally by splash, and is not subjected to the cooling action which the ring gear teeth get by being submerged in the lubricating oil. The pinion teeth are also subjected to from 5 to 7 times the number of applications of load, so that the skin temperature on the pinion teeth is maintained for from 5 to 7 times as long as it is on the ring gear. It becomes of greatest importance to use a steel and treatment which will insure maximum resistance to wear under the skin temperatures developed, and truck service tests have indicated that the martensitic structure is less adapted to this service than is a structure which contains enough austenite to develop a structure which work hardens under service loading.

It is found that the cyanided surface is considerably more stable than the uncyanided surface, and that therefore cyanide pinions are of considerable advantage in truck rear axles.

Closely approaching the cyanided pinions are those obtained by direct quenching from the carburizing temperature using steels designed for this purpose where a file hard surface can be obtained with a structure which shows considerable austenite.

One of the most satisfactory combinations for long pinion life seems to be the S.A.E. No. 6120 series, using a carburized pinion reheated in cyanide and quenched from 1630 F. in oil, together with a ring gear which is reheated and quenched from the same temperature in oil.

The importance of the stability of the surface of the pinion on a heavily loaded rear axle is indicated by the accompanying Figs. 1 and 2. Figure 1 shows a pinion made from electric furnace Krupp analysis containing approximately 4 per cent nickel and $1\frac{1}{2}$ per cent chromium. This pinion was run with a gear of the same composition, and both were double treated after carburizing to give a file hard martensitic case, having a strength of at least 300,000 lb. per sq. in. in tension. This combination of martensitic Krupp gear operating with an extreme pressure lubricant showed an average on three tests of only 53,000 pinion cycles.

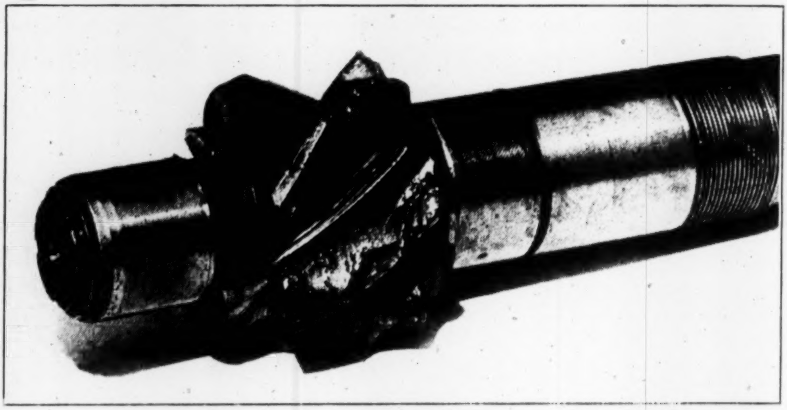

FIG. 1.—Pinion Made from Electric Furnace Krupp Steel.

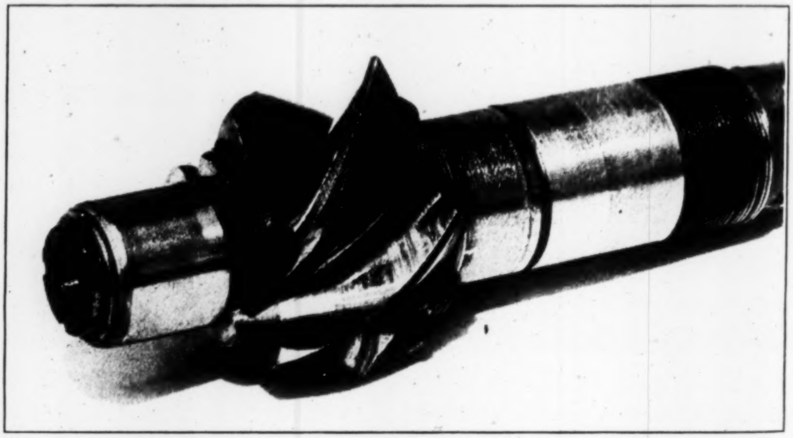

FIG. 2.—Pinion Made from S.A.E. No. 4615 Steel.

The pinion shown in Fig. 2 was made from S.A.E. No. 4615 steel, direct quenched from the carburizing box, giving an austenitic, martensitic case which, while file hard, showed a Rockwell hardness, "C" scale, of only 50, whereas the Krupp pinion shown in Fig. 1 showed a Rockwell hardness, "C" scale, of over 60.

Three S.A.E. No. 4615 pinions were run with three double-treated Krupp ring gears in the same lot as those operating with the pinions shown in Fig. 1, and gave an average life of over 130,000 pinion cycles before ring gear failure.

The failure of the Krupp pinion was due not so much to the analysis as to the treatment, and failure occurred by softening of the pinion surface followed by rapid wear on the heel of the tooth as shown in Fig. 1 and then fatiguing of the ring gear tooth on the toe end. The pinion shown in Fig. 2 showed no wear, and failure of the ring gear occurred by fatigue across the full section of the tooth, indicating no localized pressure.

It is obvious from Messrs. Almen and Boegehold's paper that in passenger car service the selection of a case-hardening steel for ring gear and pinion use is very much secondary to problems of design, etc. In heavily loaded truck service, the same conditions exist with the exception that particular care must be taken to obtain pinion surfaces which have a maximum resistance to wear after operating for a considerable period of time at skin temperatures of approximately 400 F. This requirement indicates the importance of direct quenching and, where possible, cyaniding.

Due to the difficulty in lapping the extremely hard surfaces obtained in cyaniding, it is somewhat more difficult to obtain good initial contact, but this requirement can to some degree be greatly reduced by particular care in the cutting of pinions.

It is quite obvious to those who have explored this problem fully that the use of martensitic pinions, and particularly those containing high percentages of nickel, is not recommended. It is also obvious that in order to obtain the maximum stability of the surface on the pinion, as high a carbon content as possible, consistent with normal practice, is necessary.

MR. T. H. WICKENDEN.[1]—There is one conclusion drawn by the authors that I think possibly should be limited to this one situation, namely, that there is no evidence that one alloy steel is better than another for rear axle gear application. With complicated mechanical variables in this rear axle test, the superiority of one alloy steel over another has been rather blanketed. In casually looking over data on the difference in physical property curves and heat treatment, it is evident that differences on the order of about fifty per cent can be secured by different alloys and heat treatment.

MR. A. L. BOEGEHOLD.[2]—We appreciate Mr. Peterson's reference telling how stresses in the notched fatigue test bar may be calculated, and we agree with him that grain size as it affects resistance to fatigue should be more thoroughly investigated. Presumably the suggested Neuber method of calculating stress concentration does not contemplate variations in stress concentration in different materials made into the same shaped notched bar. Table VIII shows a variation of stress concentration from 2.47 to 3.4 in the same test bar depending upon the sensitivity of the material under test. It still appears then that these values must be obtained by testing rather than by calculation. Mr. Swanger mentioned that the values above the endurance limit should be given more consideration. In certain machine structures these values of course are all important, as they are in the case of the rear axle pinion. We must design the rear axle pinion with the requirement in mind that it must withstand a certain number of maximum load applications resulting from application of low gear torque. If we designed the pinion so that stress resulting from low gear torque load were less than the endurance limit, we would not be making an economical use of our material and the pinion would be unnecessarily large. He mentioned that the "percentage strength" factor recommended by McAdam may explain the difference in the results of the different steels in the rear axle test.

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[1] Assistant Manager, Development and Research, The International Nickel Co., Inc., New York City.
[2] Metallurgist, Research Division, General Motors Corp., Detroit, Mich.

"Percentage strength" as suggested by McAdam is only another terminology for the property "notch sensitivity" which we have evaluated by means of the notched rotating-beam test. We already have the results of the notch fatigue test and they alone do not account for all the differences in behavior of the various steels in the rear axle test; therefore, I do not see how calling "notch sensitivity" by another name will supply the explanation.

Mr. Jasper mentions the importance of toughness and wear resistance and resistance to repeated shocks. We agree that there are some other properties that have to be taken into consideration in selecting gear steels besides resistance to fatigue, but we feel that resistance to fatigue is probably the most important one because failures from fatigue are more frequent in service than other types of failure. We have to take into consideration also qualities of steel which have a bearing on manufacturing processes. We must consider grain size, which determines what kind of heat treatment we can use, and by that treatment how much distortion we get. The kind of heat treatment we can use plays an important part not only in determining what kind of wear resistance we get, but also how high the maximum stresses are going to be as a result of distortion in heat treating. As a result of using a coarse-grained steel, together with certain heat treatments, more than usual distortion is likely to occur which will cause excessive maximum stresses and early failure in service or in a rear axle test. We agree with him that the Charpy test is of very little value in selecting gear steels.

Commenting on Mr. Tuckerman's discussion, I would like to say that his experience parallels ours exactly.

MR. J. O. ALMEN.[1]—I must agree with Mr. Eddy that the axle fatigue curve looks suspiciously good considering the many variables that influence gear life. However, it must be remembered that the plotted points on the fatigue curve are, in most cases, the average of three or more tests. A greater scattering would be shown if each axle were plotted separately. To illustrate, one of the points plotted on the 51,000 cycle line, Fig. 3, is the average of 3 axles that failed at 79,000, 46,000 and 30,000 cycles. Other points are averages of similarly scattered individual tests. In Fig. 7 are shown a number of averaged points that lie a considerable distance from the average curve. These points are described in detail in the paper.

Mr. Eddy says that mechanical conditions are considered to have pronounced effect on gear life. The data, I think, permits us to be definite on this point and to say that mechanical conditions have a dominant effect on gear life. That there is little difference in the alloys from which gears are made may be deduced without tests from the fact that many alloys and many heat treatments are in everyday use in production, all presumably giving satisfactory service.

For many years we have carefully watched all of the known variables in the gears tested and I do not know wherein our technique in this respect can be improved. Our observations have led us to make several definite design and machining recommendations, some of which are now coming into use. In every case where these recommendations have been followed, increased life has resulted, but we have yet to see a production axle design that has taken advantage of all the improvements that are possible.

In regard to Mr. Eddy's question on the possibility of using cheaper steel, I can see no reason for doubting that the steels he mentions would be satisfactory rear axle gear steels. In fact, Mr. Boegehold has long advocated the use of such materials. It is probable that in the majority of machine elements, the alloy used is relatively unimportant as compared to mechanical conditions producing stress concentrations. I believe cheaper steels can be used for many parts now made from the more expensive alloys.

Mr. McQuaid's comments add valuable data to that accumulated by the General Motors Research Division in that he introduces laboratory tests on truck and bus axles, a field in which our experience is limited. The destruction of tooth surfaces is not encountered in the lighter vehicles using spiral bevel gears except at high road speeds and the usual remedy is the use of extreme pressure lubricants. Our experience with cyanided surfaces agrees with Mr. McQuaid's.

I would add a remedy for high surface temperatures and resulting softening of the teeth that has not yet been mentioned. The rate of heat transfer from the gears to the atmosphere is greatly affected by the viscosity of the oil in the axle housing. Low viscosity oil reduces the stagnant oil film on the gear teeth and on the axle housing surfaces which act as insulators against heat flow from the gear teeth to the oil and from the oil to the axle housing. I would recommend the use of light oil in place of the high viscosity oils or greases in common use.

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[1] Head, Dynamics Section, General Motors Corp., Detroit, Mich.

The contact photographs shown in Fig. 11 appear to be open to misinterpretation. These photographs show ring gear teeth and Mr. McQuaid's comments are correct as regards the position of the load on these teeth. In our tests, however, failure usually occurred by fatigue of pinion teeth. Since the localization of the load at the top of the ring gear tooth is equivalent to localization of load low on the pinion tooth, Mr. McQuaid's comments do not apply to the pinion.

The sliding velocity between the mating teeth is near its minimum value when contact is made at the top of the ring gear tooth and maximum when contact is made at the top of the pinion tooth. Maximum compressive stress, however, occurs when contact is made near the top of the ring gear tooth. Pitting is most likely to occur in this region.

The gears shown in Mr. McQuaid's illustrations present a somewhat different problem in localization of stress due to deflection than is illustrated in Fig. 10 and in the tooth contact pictures, Fig. 11. Mr. McQuaid's pinions were straddle mounted and localization of load due to bearing deflections would be reduced. In a straddle mount, it is possible to avoid shifting of the tooth load, as shown in Fig. 11, by the use of a straddle bearing of greater radial rigidity than the pinion thrust bearing, thus maintaining approximately ideal contact conditions under all loads. The fact that one of Mr. McQuaid's pinions broke at the toe of the tooth was perhaps influenced by the straddle mount.

Mr. Wickenden states that a difference of about 50 per cent may be secured by different alloys and heat treatments. If that statement is based on the data shown in Table I, I think that the variations shown can be much more rationally explained as mechanical variations than as variations in alloys or heat treatments.

HIGH-SPEED FATIGUE TESTS OF SEVERAL FERROUS AND NON-FERROUS METALS AT LOW TEMPERATURES

By W. D. Boone[1] and H. B. Wishart[1]

Synopsis

The authors present a group of low-temperature high-speed fatigue tests. Two cantilever fatigue machines, developed at the University of Illinois, using 12,000 r.p.m. series motors, were used in the tests. A description is given of the machines and their operation at temperatures that varied from +80 F. to −55 F. Two types of small inexpensive specimens were tested. Unnotched specimens were used to give maximum endurance limits for the materials, while notched specimens were used to show the effect of stress concentration.

Endurance limits of duralumin, brass, gray cast iron, meehanite cast iron, cold-drawn steel, and rail steel were determined for unnotched and notched fatigue specimens for temperatures of +80 F., +10 F., −20 F., and −40 F.

In general, as the temperature was decreased the endurance limits of the metals increased. The stress concentration factors showed no consistent change.

Low-temperature tests on the physical properties of metals have been somewhat neglected in the past—possibly due to little demand for information in this field and also because of the expense of running such tests. Now, with industry developing processes requiring the exposure of metals to sub-zero temperatures, the growth of aviation, and railroads laying and maintaining track in frigid zones, demand for information about the behavior of metals in this field is increasing.

While most of the low-temperature work[2] on metals has been confined to impact and tension tests, the work presented in this paper has been confined to a study of the fatigue properties of metals with temperature change. The data reported in this paper give the endurance limits of unnotched and notched specimens of ferrous and non-ferrous metals at various low temperatures as determined by high-speed fatigue machines.

The tests were run in the cold room of the Material Branch, Wright Field, Dayton, Ohio. Acknowledgment is hereby made to the War Department and Mr. J. B. Johnson for its use.

Space in the cold room during these tests was limited and the time of operation of the cold room at each temperature was limited, so the use of high-speed fatigue machines was very desirable. The two machines occupied a floor space only 2 ft. square and their speed of operation permitted determination of an endurance limit in about one-fourth the time required with the rotating-beam machines running at 1800 r.p.m.—a common speed for such machines.

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[1] Special Research Assistant, Materials Testing Laboratory, University of Illinois, Urbana, Ill.

[2] See bibliography appended to this paper, p. 151.

The cold room was 10 ft. square by 8 ft. high enclosed by 12-in. cork walls. A 12-ton Wittenmeier carbon dioxide compressor system maintained the temperature of the room at any desired value from +80 F. down to −50 F. Other physical test equipment in the cold room had no appreciable effect upon the operating temperature of the high-speed fatigue machines which were used in this test. The room temperature was regulated within ±3 deg Fahr.

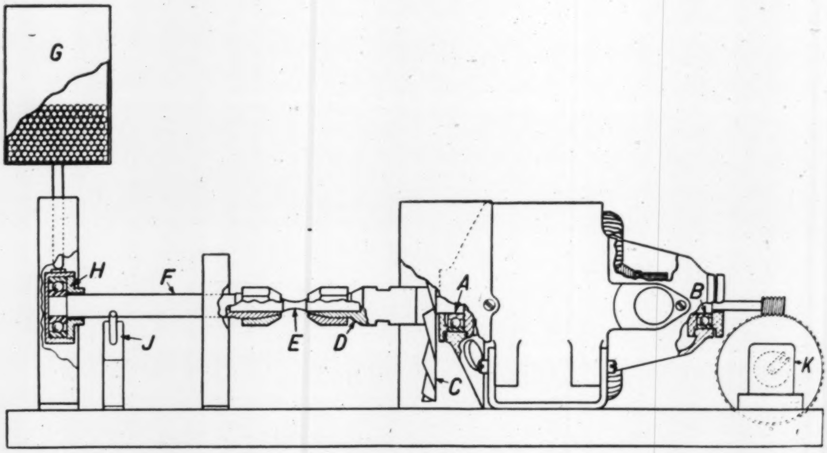

FIG. 1.—High-Speed Fatigue Machine.

Series-wound electric motors drove the high-speed fatigue machines at 12,000 r.p.m. This type of motor had good operating characteristics for use on a fatigue machine. Under constant load it runs at constant speed, or its speed can be held at any value from 3000 to 15,000 r.p.m. by a simple adjustment of a rheostat. In the laboratories of the University of Illinois, these machines have been operating more than a year with no repairs and little apparent wear.

Figure 1 shows a diagram of the fatigue machine. Ball-bearings *A* and *B* of the motor support the rotating element of the machine. Blower *C* on the motor shaft circulates air to cool the machine. Collet *D* on the shaft grips the specimen *E*. Load arm *F* with a similar collet exerts a bending moment on the specimen *E* as load cylinder *G* bears upon load bearing *H*. When the specimen fails, load arm *F* drops, tripping cut-off switch *J* and stopping the machine. The cycles of stress are recorded by counter *K* driven through a 100 to 1 worm and gear reduction on the rear motor shaft extension.

For low-temperature tests the oil in the bearings was thinned with naphtha as needed. The motors and load bearings operated very satisfactorily at low temperatures.

The stress in the specimen is computed from the cantilever-beam formula. The distance from the center line of the load cylinder to the critical section of the specimen, the moment arm of the load, is adjusted to 4.5 in. by a trammel gage as the specimen is mounted in the machine. The load consists of the weight of the load cylinder *G* plus a constant for the effective weight of the load arm *F* at the center line of the load.

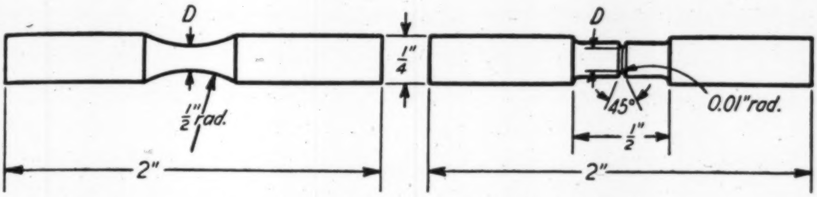

FIG. 2.—High-Speed Test Specimens.

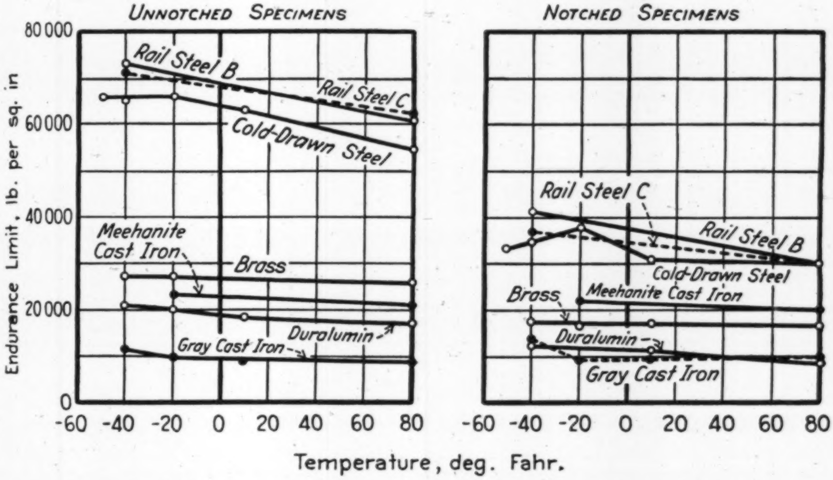

FIG. 3.—Variation of Endurance Limits with Temperature Change.

TESTS AND TEST SPECIMENS

Fatigue tests were made on specimens of duralumin, brass, gray cast iron, meehanite, cold-drawn steel, and rail steel. Most of the metals were tested at four different temperatures: +80 F., +10 F., −20 F., and −40 F. The cold-drawn steel in addition to these temperatures was tested at −50 F.

Tests of unnotched and notched specimens of each metal were run at each temperature. The test results from the unnotched specimens were used as a standard with which to compare test results from the notched specimens and also to determine the effect of temperature change upon the metal as a whole. The notched specimens were used to study the effect of low temperatures on the effective stress concentration at the bottom of a notch.

TABLE I.—CHEMICAL COMPOSITION, PER CENT, AND HEAT TREATMENT OF METALS TESTED.

Duralumin	4.24 Cu, 93.56 Al, 0.59 Fe, 0.56 Mg, 0.24 Si, 0.71 Mn, 0.10 Ca. Tested as received.
Brass (cold drawn)	62 Cu, 35 Zn, 3 Pb. Annealed 45 min. at 650 F., cooled in furnace.
Gray cast iron	3.25 C, 0.60 Mn, 0.46 P, 0.096 S, 1.1 Si. Annealed 30 min. at 1040 F., cooled in furnace.
Meehanite cast iron	3.07 C, 0.9 Mn, 0.15 P, 0.08 S, 1.26 Si. Tested as received.
Cold-drawn steel	Tested as received.
Rail steel B	0.72 C, 0.75 Mn, 0.03 P, 0.023 S, 0.15 Si. Tested as received.
Rail steel C	0.75 C, 0.76 Mn, 0.024 P, 0.029 S, 0.19 Si. Tested as received.

TABLE II.—ENDURANCE LIMITS AND STRESS-CONCENTRATION FACTORS AT VARIOUS TEMPERATURES.

Specimen Temperature, deg. Fahr.	Duralumin	Brass	Gray Cast Iron	Meehanite Cast Iron	Cold-Drawn Steel	Rail Steel B	Rail Steel C
Endurance Limit,[a] lb. per sq. in.—Unnotched Specimens							
+80	17 000	26 000	9 000	21 000	55 000	61 000	62 000
+10	18 500	.....	9 000	.....	63 000	.....	.....
−20	20 500	27 500	9 500	23 000	66 000	.....	.....
−40	21 000	27 000	11 500	.....	65 000	73 000	72 000
−50	.....	.....	.....	.....	66 000	.....	.....
Endurance Limit,[a] lb. per sq. in.—Notched Specimens							
+80	9 000	17 000	10 000	20 000	30 000	30 000	30 000
+10	12 000	17 500	10 000	.....	31 000	.....	.....
−20	.....	16 500	9 500	22 000	38 000	.....	.....
−40	13 000	17 500	13 500	.....	35 000	41 000	37 000
−50	.....	.....	.....	.....	33 000	.....	.....
Stress-Concentration Factor = $\frac{\text{Endurance Limit Unnotched}}{\text{Endurance Limit Notched}}$							
+80	1.89	1.53	0.90	1.05	1.83	2.03	2.06
+10	1.54	....	0.90	....	2.03	....	....
−20	....	1.67	1.00	1.05	1.74	....	....
−40	1.62	1.54	0.85	....	1.86	1.78	1.95
−50	....	....	....	....	2.00	....	....

[a] Endurance limit based on 50,000,000 cycles for duralumin and brass; other endurance limits based on 10,000,000 cycles.

Figure 2 shows the two types of specimens used in the tests. The diameter *D* at the critical section of the specimens depended upon the material—metals with low endurance limits having large diameters and *vice versa*. The variation of the diameters was necessary in order to keep the load on the specimens within a feasible range.

The temperatures of the specimens were measured with a small thermocouple. The specimens ran approximately five degrees warmer than the temperature of the cold room. All temperatures mentioned in this paper are specimen temperatures.

RESULTS

Table I gives the chemical composition and heat treatment of the metals tested.

Table II gives the endurance limits of specimens tested at the various temperatures, and the stress-concentration factors of the notch as determined by the ratio of the endurance limits of the unnotched specimens to those of the notched specimens. For the ferrous metals the endurance limits are based on 10,000,000 cycles of reversed flexure, while the endurance limits for the non-ferrous metals are based on 50,000,000 cycles. Figure 3 gives a graphical presentation of the data from Table II. In general, the endurance limit of a metal represented by each point on the curve was determined from tests of five or more specimens.

CONCLUSIONS

1. All of the metals tested showed an increase in endurance limit as the temperature was decreased from +80 F. to −50 F. The cold-drawn steel, however, after reaching −20 F. showed no further increase in endurance limit.

2. The stress-concentration factor as determined by the ratio of endurance limits of the unnotched to the notched specimens showed no consistent change with decreasing temperatures. There was enough variation in the stress-concentration factor to indicate that further investigation would be of interest.

3. The high-speed fatigue machines used in these tests operated satisfactorily at low temperatures. These machines proved to be particularly valuable in shortening the time of the tests, since time was an important factor in the operation of the cold room.

It would be of interest to investigate the effect of over-stressing in the fatigue testing of metals at low temperatures. The study of the probable damage line method due to overstressing could be carried out using the method employed by H. J. French.[1]

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DISCUSSION

Mr. R. E. Peterson[1] (*presented in written form*).—Failures of railway apparatus parts at sub-zero temperatures have not been clearly understood in the past. However, recent experimental results showing sharp decrease in impact values at sub-zero temperatures and the slight increase of endurance values at the same temperatures have enabled engineers to interpret these failures properly and to work out the proper solutions.

As a matter of scientific interest the following point may be brought out. The tensile strength is known to increase with decreasing temperature; in tests by Gruschka[2] the increase was 50 to 150 per cent at −200 C. Does the endurance limit increase proportionally, that is, is the endurance ratio constant with decreasing temperature?

The results of the grooved specimen tests may be of value for correlation with other similar tests. In this connection it would be of value to know dimension "*D*" for the various materials tested.

The authors are to be congratulated on the further development of high-speed fatigue testing which seems certain to find a place in materials testing laboratories.

Mr. R. L. Templin[3] (*presented in written form*).—This paper is of special interest in that it furnishes definite information on the effect of low temperatures on the endurance limits of several materials. Although there has been good reason for believing that endurance limits increased or at least did not decrease as the temperature decreased, the authors have proved that the materials they have tested actually have higher endurance limits at temperatures below room temperature than at room temperature.

Tension tests at 70 and −110 F. on duralumin having a composition similar to that reported in the paper were reported several years ago.[4] These tests showed much less effect on the tensile strength than the authors report for the endurance limit. The fact that the tests made by Messrs. Boone and Wishart were carried out to only 50,000,000 cycles may partially account for the large increase in the value reported for a temperature of −40 F. over the value for +80 F.

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[1] Manager, Mechanics Division, Research Laboratories, Westinghouse Electric and Manufacturing Co. East Pittsburgh, Pa.

[2] G. Gruschka, "Zugfestigkeit von Stählen bei tiefen Temperaturen," *Forschungshelft No. 364*, Vereines Deutscher Ingenieure Verlag, Berlin (1934).

[3] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[4] R. L. Templin and D. A. Paul, "The Mechanical Properties of Aluminum and Magnesium Alloys at Elevated Temperatures," Symposium on Effect of Temperature on the Properties of Metals, published jointly by the American Society for Testing Materials and The American Society of Mechanical Engineers, p. 290 (1931). Symposium available as separate publication.

On the basis of tests made at low temperatures and at elevated temperatures on material similar to the duralumin tested by the authors, endurance ratios for various temperatures have been computed. These ratios have been plotted against temperature as given in the accompanying Fig. 1. It will be noted that the endurance ratio is about 0.35 at −40 F. and decreases to about 0.29 at room temperature and then the curve seems to become asymptotic to some such value as 0.25.

It is also interesting to note that the stress-concentration factor for the 45-deg. notch is practically the same at low temperature as at normal room temperature.

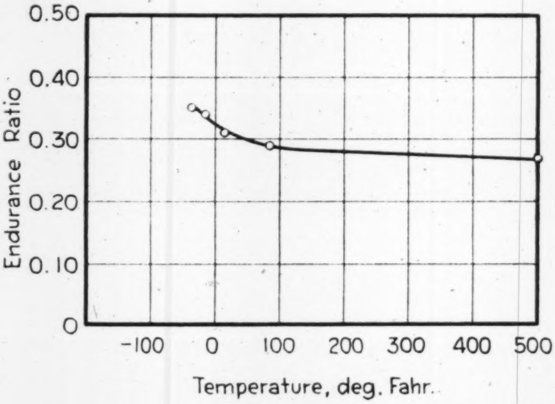

FIG. 1.—Endurance Ratio *Versus* Temperature (17S-T).

MR. J. T. MACKENZIE.[1]—I should like to ask the authors whether they attach any significance to the sudden drop in the value of the stress-concentration factor for gray cast iron from −20 F. to −40 F.

MR. E. S. DIXON.[2]—I was impressed with the endurance limit for Meehanite cast iron as compared with that for gray cast iron. I should like to like to ask the authors whether cast irons alloyed with either nickel or molybdenum, which are being commonly used, would not give better or equivalent values as that shown for Meehanite cast iron? Do the authors explain this difference in strengths as due to difference in hardness, structure, or a result of the manufacture of this particular brand of cast iron?

MR. W. D. BOONE.[3]—In answer to Mr. Peterson, we did not determine the tensile strengths and so could not compute the ratios of endurance limits to tensile strengths, but Mr. Templin does supply some values. The value of the dimension "*D*" was varied for the various materials. To maintain a convenient range of bending moment on the specimens, the dimension was made such that $D = \frac{4.5}{\sqrt[3]{S}}$ (approximately), where S was the stress at the endurance limit.

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[1] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.
[2] Metallurgist, The Texas Company, Port Arthur, Tex.
[3] Special Research Assistant, Materials Testing Laboratory, University of Illinois, Urbana, Ill.

We attach no significance to the drop in value of the stress-concentration factor for gray cast iron at −40 F., which Mr. MacKenzie questions. The tests made were too few to draw any conclusion concerning this point.

Further tests on cast irons and alloy cast irons such as Mr. Dixon suggests would be quite valuable. Nickel and molybdenum cast irons would probably compare very favorably with Meehanite cast iron. The high endurance strength of the Meehanite is probably caused by an improved structure resulting from the manufacturing process used.

THE ROTATING-WIRE ARC FATIGUE MACHINE FOR TESTING SMALL-DIAMETER WIRE

By J. N. Kenyon[1]

Synopsis

A stress-reversal fatigue machine developed on the principle of a rotating wire bent to arc curvature is being used to test small-diameter wire. Resonant vibrations or standing waves are dampened in an oil bath. The specimen automatically assumes the form of a circular arc by the elimination of flexural shear. Test results so far obtained give satisfactory values for small-diameter wire.

Intensive investigations have shown that the fatigue limit of metals is to a large extent controlled by surface condition. Surface decarburation, scratches, and other imperfections may in some instances reduce the fatigue limit of a product to less than one-half(**9**)[2]. Small-diameter wire due to the small cross-section is especially susceptible to the effect of the surface condition and is a problem not so easily controlled in the metallurgical practice. This fact is of added importance when it is considered that small-diameter wire has wide commercial application in the manufacture of cable and of other fabrications subjected in service to pulsating stresses. While fatigue tests on wire may not always give a direct indication of the endurance properties of the fabricated wire product, they *do* give a direct indication of the quality of the material entering into the fabrication.

As the endurance properties of a material are to a large extent controlled by surface condition, the unavoidable inroad of corrosion is serious. Metallic coatings are being extensively used for corrosion prevention and their effect on the fatigue limit of a material is still a field for extended investigation.

Data have recently been published to show that there is apparently no relation between the fatigue limit (endurance properties) of a wire and its other physical properties with the possible exception of the reverse bend test(**3**). In view of these facts, and the direct relation of fabricated wire products to the problem of public safety, it seems essential that relatively simple yet accurate test methods be developed for determining the service qualities of these materials.

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[1] Testing Engineer, Civil Engineering Testing Laboratories, Columbia University, New York City.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 163.

SURVEY OF OTHER TEST METHODS

The most obvious method of making fatigue tests on small-diameter wire is by pulsating tension load application. This method has offered some difficulties in accurate calibration and, until quite recently, in the gripping of the test specimens(7,8,9). Valuable work is being done on wire at the National Bureau of Standards with Haigh alternating-stress machines but it is uncertain as to whether these machines are adaptable to the application of a load under 500 lb.

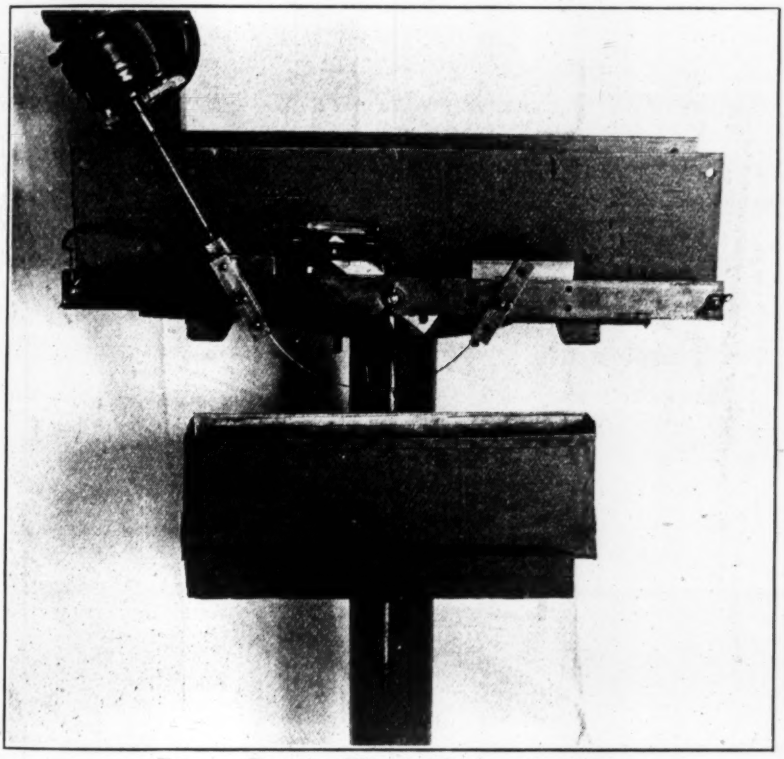

FIG. 1.—Rotating-Wire Arc Fatigue Machine.

Lindeberg of Germany has devised a tension-fatigue machine wherein pulsations are obtained by impressing an alternating current on a d.c. motor. The method is being used to test 1.00 to 1.37-mm. (about 0.04 to 0.054-in.) diameter wire(5).

One of the first machines for wire based on the stress-reversal method was developed by Shelton at the Bureau of Standards. It is of the rotating-beam type of construction and is suitable for testing wire of constant cross-section if the length of the specimen gives weight sufficient to secure center or near-center breaks. Resonant vibrations are avoided by speed control(6,9).

de Forest and Hopkins have made tests on wire by rotating the specimens under tension while bent to a circular arc around a sheave(2).

Templin has a method for testing wire and small-diameter rope in 50-ft. lengths by subjecting the specimens to a combined tension and bending stress(10).

A machine adapted to the testing of small-diameter wire has been designed by Haigh and Robertson at the Brunton Laboratories, Scotland. The specimen is essentially a rotating wire bent to strut form by end thrust. Stress computations are based on the Euler formula. Results of fatigue tests on 0.080-in. diameter wire have recently been published(3).

Friedman of Germany has developed a machine for subjecting wire to alternate bending. The specimens are 1.5 to 3.5 mm. (about 0.060 to 0.14 in.) in diameter. The method is being used to determine the endurance qualities of wire welds(1).

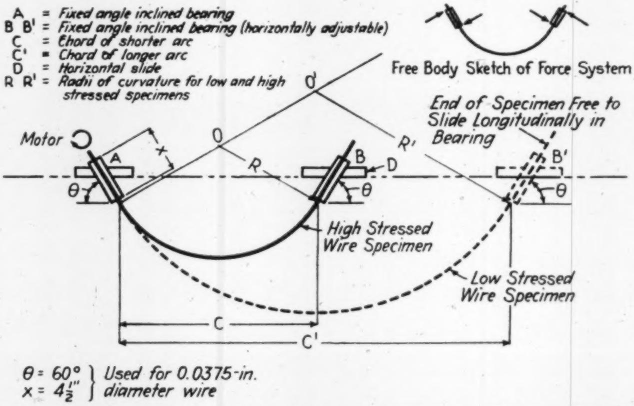
A = Fixed angle inclined bearing
B B' = Fixed angle inclined bearing (horizontally adjustable)
C = Chord of shorter arc
C' = Chord of longer arc
D = Horizontal slide
R R' = Radii of curvature for low and high stressed specimens
Free Body Sketch of Force System
O'
End of Specimen Free to Slide Longitudinally in Bearing
Motor
O
x
R
R'
A
B
D
B'
θ
θ
θ
High Stressed Wire Specimen
Low Stressed Wire Specimen
C
C'
θ = 60°
x = 4½"
Used for 0.0375-in. diameter wire

FIG. 2.—Diagrammatic Sketch of Rotating-Wire Arc Fatigue Machine.

THE ROTATING-WIRE ARC FATIGUE MACHINE

The rotating-wire arc test method was developed to determine the effect of different metallic coatings on steel wire in which results of an empirical or comparative nature only were expected. It was found, however, that fatigue values could be obtained with a high degree of reproducibility. Fatigue limits obtained on steel wire with the usual commercial surface condition compare favorably with results on similar materials obtained by other investigators(3,6). In that the principle appears to be mathematically sound, this method is considered adaptable to the testing of small-diameter wire.

The machine consists essentially of two inclined babbitt metal bearings holding the wire test specimen in curved form and in a vertical plane.

The lower end of the curve dips in an oil bath. One end of the wire is attached to a small electric motor and the other end is free to adjust itself longitudinally in the inclined bearing (Fig. 1). A Veeder counter is attached to the motor. As the specimen rotates, it automatically eliminates flexural shear at the free end and assumes the form of a circular arc. Both bearings are inclined at a definite fixed angle and the different radii of curvature are obtained by horizontal adjustment of one inclined bearing. Straight test specimens are required. In Fig. 1 the oil container is lowered to show the wire test specimen and a *trip* whereby a mercury switch is tilted and opened when the specimen breaks.

Figure 2 is a diagrammatic sketch of the arc machine. Different radii of curvature are secured by horizontal adjustment of the right inclined bearing. Stress computations are based on direct measurement of the horizontal chord, C, wherein the ends of the horizontal slides, D, are used as convenient reference points.

Stress Determinations.—Stress determinations are based on the arc formula:

$$\text{Extreme fiber stress, lb. per sq. in.} = \frac{EI}{RZ} = \frac{Ed}{2R} = \frac{Ed}{C}\sin\theta$$

where E = modulus of elasticity,
Z = section modulus,
d = diameter of wire specimen,
R = radius of survature,
θ = angle of inclined bearing with the horizontal, and
C = horizontal chord = $2R \sin \theta$.

A relatively low viscosity oil was found to be satisfactory for the oil bath. The specimen rotates in the oil with scarcely visible movement and is not noticeably affected by speed variation. An energy input determination showed that the drag effect of the oil on an 0.038-in. diameter wire amounts to a torque of 0.0045 in-lb. and corresponds to a shear stress of 435 lb. per sq. in. The torque due to friction in the bearings was too small for accurate determination. Shelton also found bearing friction to be of a small order (6). Lehmann found no effect on the fatigue limit from testing a steel specimen in machine oil (4).

The tendency of the wire to sag due to its own weight is found to be negligible since a double exposure on a sheet of blueprint paper with the wire arc in an upward and downward position respectively showed no measureable change in curvature. Breaks may occur at any point along the arc but they seldom occur at the ends of the bearings. The wire acts as its own shaft and does not undergo appreciable wear in the bearings. As an example of this fact, a copper-beryllium wire specimen was only slightly worn after 100,000,000 stress reversals.

It is found that the test method is equally applicable to soft materials such as annealed copper. However, if these materials are not perfectly straight there will be some vibration in the oil bath but this vibration usually disappears as the test proceeds.

The arc machine would seem to offer the following advantages:

1. Nominal cost,
2. Ease and simplicity of operation,
3. No gripping problem or bearing failures,
4. Vibrations dampened in an oil bath,
5. Fatigue breaks not confined to one point, and
6. Self calibrating due to the specimen assuming the arc form (an accurate determination of the modulus of elasticity is assumed).

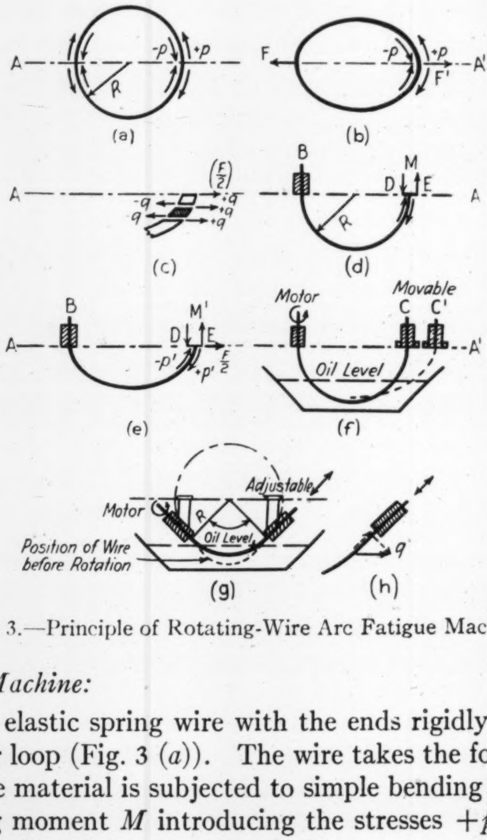

FIG. 3.—Principle of Rotating-Wire Arc Fatigue Machine.

Principle of the Machine:

Consider an elastic spring wire with the ends rigidly attached and so bent to a circular loop (Fig. 3 (*a*)). The wire takes the form of an arc of a circle wherein the material is subjected to simple bending stresses only and with the bending moment M introducing the stresses $+p$ and $-p$ on the outside and the inside of the arc respectively.

Assume that equal and opposite forces, F and F', are applied to the wire along the axis, $A—A'$ (Fig. 3 (*b*)). The wire will be distorted from its circular form and will also have flexural shearing stresses, $+q$ and $-q$, across horizontal sections of the material (Fig. 3 (*c*)).

Let us now eliminate the wire above the axis, $A—A'$, in Fig. 3 (*a*), hold one end rigidly at B, introduce the rigid line DE and retain the moment by introducing forces at D and E (Fig. 3 (*d*)). We now have the same equilibrium condition as shown in Fig. 3 (*a*). The rigid line DE indicates that the tangent of the arc at the free end is maintained in a vertical direction as it was in Fig. 3 (*a*) and (*b*).

Let the wire be eliminated above $A—A'$ in Fig. 3 (*b*) and hold one end rigid as at B in Fig. 3 (*e*). Again maintain the direction of the tangent at the free end; that is, introduce the rigid line DE. By applying the proper external moment M' and force $F/2$ the condition is equivalent to that of Fig. 3 (*b*). Under this condition the moment M' is larger than the M of Fig. 3 (*d*) and the fiber stresses, $+p'$ and $-p'$, at the free end are also larger than those existing in the case of Fig. 3 (*d*). It may also be noted that the fiber stresses are not uniform throughout the length of the curve but the internal forces are in equilibrium with the external force system consisting of M' and $F/2$.

If the end of the curved wire is free to move in a horizontal direction, that is, if no horizontal restraint is afforded, the force $F/2$ will be eliminated. The external force system will now consist of an end moment only and the internal stresses existing in the curved wire indicated in Fig. 3 (*e*) will not be in equilibrium with such an external system. For moment applied at the ends, the curvature will be circular $\left(\frac{1}{R}=\frac{M}{EI}\right)$ and the stresses uniform throughout. With complete freedom of movement horizontally at one end, therefore, no horizontal force $F/2$ can be developed and the distortion must be that of "simple bending"; that is, the arc of a circle. The condition will be the same as that of Fig. 3 (*a*) and (*d*).

The wire test specimen is held in babbitt metal bearings maintained in a vertical position. It is rotated by a motor and forms its own shaft (Fig. 3 (*f*)). One bearing is fixed in position and the other mounted on a carriage with freedom of movement along axis $A—A'$ (suspending the bearing will also eliminate any horizontal forces at the free end). If the movable bearing is started in a position C', it will move to position C to relieve flexural shear and allow the specimen to assume the circular curvature as explained above. In other words, with the apparatus designed to eliminate any force $F/2$ at the free end, no shear can be developed and hence only bending stress will exist.

Flexural shear may be effectively eliminated from the test specimen by tilting the babbitt metal bearings to some fixed angle from the horizontal position (Fig. 3 (*g*)). Assume that the two bearings are fixed horizontally and that the right end of the specimen may slide freely through the right bearing. If the specimen assumes the position indicated by the broken line, the flexural shear will make it tend toward the circular arc form indicated by the solid line. The right bearing in this arrangement is restrained from moving along the horizontal axis, since the rotating wire will slide in the bearing due to the longitudinal component of the flexural shear (Fig. 3 (*h*)). That is, the freedom of movement in this case is afforded by allowing the wire to slide longitudinally in the bearing. In this setup the wire is not suspended as shown in the previous diagrams. The tendency to sag due to the very small weight of the wire specimen is largely offset by flexural shear and by the buoyancy of the oil bath, and has been found to be negligible.

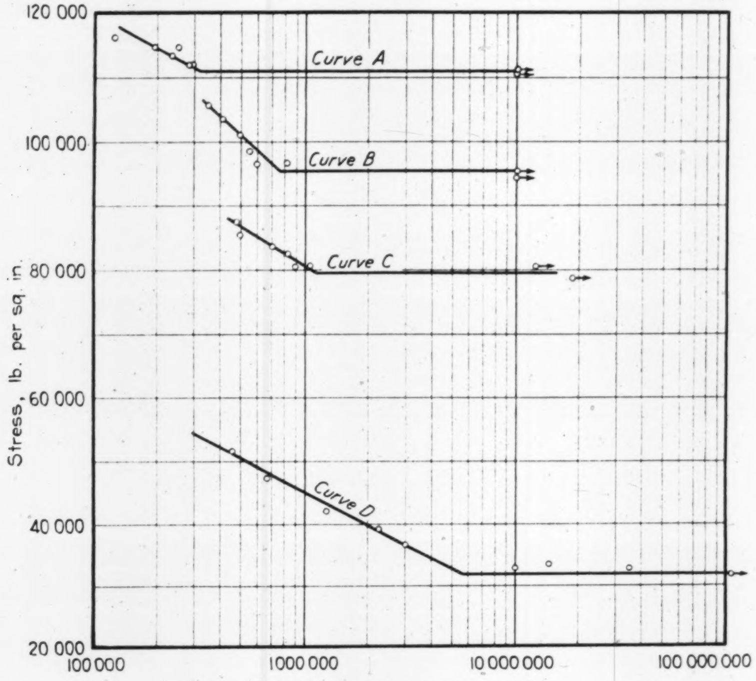

FIG. 4.—Rotating-Wire Arc Fatigue Test on Wire.

TEST RESULTS

The arc machine is of recent development and has been used largely to test materials of an experimental nature. As a result there is a limited amount of data suitable for this publication. The following table is a summary of fatigue values obtained on commercial materials:

	Material	Tensile Strength, lb. per sq. in.	Fatigue Limit, lb. per sq. in.	Ratio, Fatigue Limit to Tensile Strength
Curve A[a].....	piano wire (C 0.85 to 0.88, Mn 0.30 to 0.35)	374 000	111 000	29.7
Curve B[a].....	cold-drawn steel wire (C 0.66, Mn 0.80)	282 000	95 000	34.0
Curve C[a].....	cold-drawn steel wire (C 0.60 to 0.65, Mn 0.75 to 0.80)	275 000	79 500	29.0
Curve D[a].....	copper (2.25 Be) wire (untreated)	132 500	31 500	24.0

[a] See Fig. 4.

The above fatigue values were obtained on an 0.0375-in. diameter wire with the usual commercial surface condition. The regularity of the graphs (Fig. 4) are indicative of the degree of reproducibility. The values for steel are in very good agreement with results on more or less similar materials tested by Shelton(6) and Gill and Goodacre(3).

SUMMARY

It should be pointed out that whereas the Shelton machine is essentially a constant stress test method, the rotating-wire arc machine is a constant strain method. The latter method requires the modulus of elasticity for stress computation.

A careful measurement of curvature has shown that the wire arc varies from the computed value in the order of one per cent. The bearings cannot permit freedom of rotation and still function as perfect tangents. As a result there is apparently a small decrease in stress from the computed value at the ends of the bearings and this may account in large part for so few fatigue breaks at these points. More work is being done to clarify this behavior and to determine the effect of different inclinations of the bearings.

In view of the results so far obtained it is felt that the rotating-wire arc machine has important and fundamental characteristics and that the principle presented should open up a new field for testing small-diameter wire.

Acknowledgments.—Appreciation is due Colin G. Fink for his sympathy and encouragement during the development of the rotating-wire arc idea, and also to W. J. Krefeld and J. C. Rathbun.

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(3) E. T. Gill and R. Goodacre, "Some Aspects of the Fatigue Properties of Patented Steel Wire," *Journal*, Iron and Steel Inst. (London), Vol. 130, No. 2, p. 293 (1934).

(4) G. D. Lehmann, "The Variation in the Fatigue Strength of Metals when Tested in the Presence of Different Liquids," *Reports and Memoranda No. 1054*, Great Britain Aeronautical Research Committee, p. 2 (1926).

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(7) W. H. Swanger and R. D. France, "Effect of Zinc Coatings on the Endurance Properties of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 430 (1932).

(8) S. M. Shelton and W. H. Swanger, "Fatigue Tests on Galvanized Wire Under Pulsating Tension Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 348 (1933).

(9) S. M. Shelton and W. H. Swanger, "Fatigue Properties of Steel Wire," National Bureau of Standards *Research Paper No. 754*, Vol. 14, p. 17 (1935).

(10) R. L. Templin, "The Fatigue Properties of Light Metal Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 371 (1933).

DISCUSSION

MR. L. W. HOPKINS.[1]—Several years ago Mr. de Forest and I worked along lines similar to those of Mr. Kenyon to determine the fatigue properties of wire. We were very much bothered by indeterminate stresses caused by vibration. Our apparatus consisted of a bench lathe. We had little speed adjustment nor did we immerse the wire in a dampening medium so our experiments were not directly successful but they did lead to the development of apparatus described before the Society in 1932.[2]

Stress concentrations at the jaws of the vise or grip provide the stumbling block for most experiments of wire fatigue tests and Mr. Kenyon seems to have overcome this difficulty. One other important consideration lies in the amount and type of straightening necessary to prepare the specimen. If the wire must be straightened its elastic properties are changed. Professor Moore has mentioned a case in which fatigue specimens had been subjected to ten million reversals without failure and that subsequent increase in the applied stress and further application of cyclic loading indicated that the original test had work-hardened the specimens and increased the endurance limit. The same thing happens when wire is straightened or massaged. We have noted some cases where the endurance limit was raised 20 per cent by merely massaging the wire previous to the fatigue test.

Mr. Kenyon's apparatus shows with ours the advantage of testing a specimen having a higher length to diameter ratio. This permits the simultaneous testing of a higher gross quantity of material and tends to keep down the effect of minor stress concentrations which have little practical consideration.

[Mr. Hopkins then showed a series of slides illustrating the development of the endurance test for wire described in A.S.T.M. Proceedings *loc. cit.* and a miniature apparatus designed to make endurance tests of wire 0.008 to 0.020 in. in diameter.]

MR. R. L. TEMPLIN.[3]—I too would be interested to know whether it is necessary to use straightened samples in this test, or take samples in the coiled condition as they come in the wire as usually received? We have had quite a little difficulty in making tests with the Shelton type machine when the specimens are not straight, and as Mr. Hopkins has pointed out, you always introduce some questionable elements when you perform a straightening operation on the wire. We sometimes overcome this difficulty by removing the coiling reel an appreciable distance from the draw bench and getting our samples before the wire is coiled, thus avoiding straightening troubles.

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[1] Materials Engineer, American Chain Co., Inc., Bridgeport, Conn.

[2] A. V. de Forest and L. W. Hopkins, "The Testing of Rope Wire and Wire Rope," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 398 (1932).

[3] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

MR. J. N. KENYON.[1]—Attempts have been made to test wire taken directly from the coil and as one might expect the results have been erratic as the concave side of the wire is subjected to entirely different stresses than the convex. A selected straight specimen will naturally give much more satisfactory results. This leads to the question as to whether the specimen should be commercial wire taken direct from the coil or one that has not been subjected to this coiling treatment. Tests along this line would be very interesting.

MR. H. F. MOORE.[2]—Mr. Kenyon has done something which, at first, struck me as almost unbelievable, but after studying it, it struck me as reasonable. In the machine, is the arc of the wire a circle from bearing to bearing?

MR. KENYON.—Approximately, from end to end of bearings.

MR. MOORE.—So there would be the same stress at the edge of the bearing as in the center of the wire?

MR. KENYON.—Theoretically the curve taken by the wire is very close to that of the circular arc with a slight tendency toward the catenary due to the weight of the wire. Measurements have been made, as described in the paper, to show that this deviation is very slight and the fact that a large percentage of breaks occur well away from the center would lead one to believe that it is of no practical consequence.

MR. MOORE.—May I point out that Mr. Kenyon seems to have obtained a result which experimenters in fatigue have been seeking for a long while, but usually they have been working, not with bearings around wire, but with collets which grip the wire. For the most part they have been working on high-strength steels, and they have had a great deal of trouble with fractures at the collet jaws. I wonder if the reason Mr. Kenyon got failures away from the bearing was because the wire ran in a lubricated bearing rather than being gripped in a collet.

MR. KENYON.—Probably the reason that breaks occur at the bearings is due to vibration. A vibrating wire is subjected to excessive bending at the points held rigid, which condition is not true at nodal points or at any other points along the wire. This would be especially true if the wire specimen were rotated at certain critical speeds commensurate with its natural frequency.

The oil bath exerts a dampening effect which is so effective that no trouble with vibration or bearing breaks was experienced even at critical speeds. The author obtained bearing breaks before he resorted to the oil bath.

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[1] Testing Engineer, Civil Engineering Testing Laboratories, Columbia University, New York City.
[2] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

INFLUENCE OF TIME ON CREEP OF STEELS

By A. E. White,[1] C. L. Clark[1] and R. L. Wilson[2]

Synopsis

This paper presents the results from extended-time creep tests conducted at 1000 F. on a plain carbon steel of the S.A.E. 1015 type and a chromium-silicon-molybdenum steel. Certain of the stresses for the alloy steel were so selected that fracture would result at various periods of the test, thus producing the complete time-elongation curves. It should be emphasized that these larger stresses are considerably greater than any which would normally be used in commercial practice. The fractured specimens were then subjected to metallographic, X-ray and hardness examinations to determine the extent to which the structural and deformation characteristics were dependent on the time required for rupture.

The results presented indicate the creep rates, and thus the reported creep stresses, to be influenced by the length of time the creep tests are extended. While not affected to the same degree, with both steels lower creep stresses were obtained from the 500-hr. test than from the 1000-hr. test and were therefore more comparable to those obtained from the greatly extended tests.

The structural and deformation characteristics were found to be influenced by the time required for rupture. This was evident from the metallographic, X-ray and hardness examinations. The observed differences may be explained either on the assumption that the mechanism of deformation is different in the short-time tension test than in the creep tests or that, with the creep specimens, the deformation characteristics were changed because of the time at temperature.

Introduction

In determining the creep characteristics of metals at elevated temperatures, there has always been a question as to the length of time these tests should be conducted. The ideal procedure would be, of course, to maintain specimens of the given materials under the desired operating stresses and temperatures for time periods equivalent to the commercial life desired. For the majority of cases, however, this is not possible as the erection of the apparatus could not be sufficiently delayed to permit completion of the creep tests. Moreover, if such a procedure were followed, the amount of available creep data would be extremely small and the laboratory phase of the work would consequently fall considerably behind commercial progress, thus making the selection of the proper metals for high-temperature service even more a matter of chance than at present.

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[1] Director, and Research Engineer, respectively, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

[2] Metallurgical Engineer, The Timken Steel and Tube Co., Canton, Ohio.

Since the creep tests are seldom if ever conducted for time periods approaching the actual life desired, it is necessary arbitrarily to select the time these tests are to be conducted. Early attempts were made to develop short-time tests for indicating the creep characteristics and as a result the methods of Barr and Bardgett, Hatfield, and Pomp were advanced, as well as the use of the proportional limit as obtained from the short-time tension tests. None of these methods has been very successful and their use has been confined largely to the laboratories of their sponsors. Present-day creep tests are extended for periods ranging from a few hours to several thousand hours, with the vast majority probably falling within the range of 500 to 1000 hr.

Irrespective, however, of the time involved in the tests, it is customary to report the results in terms of the stresses required for creep rates of 1 per cent in either 100,000 or 10,000 hr. These results are obtained on the assumption that a creep rate of 0.01 per cent per 1000 hr. is equivalent to 1.0 per cent per 100,000 hr. and 0.10 per cent per 1000 hr. equivalent to 1.0 per cent per 10,000 hr. Certain groups are now advocating that the results be expressed in terms of either inches per inch per hour, or per cent per hour. This procedure has the advantage that, through extrapolation, the results can readily be expressed in any given units. The importance of the time factor is not eliminated, however, and the time period at which the rate is computed should be stated.

The question then arises as to the influence of the duration of the test on these reported creep values. Very little published information is available on this subject even though two valuable contributions[1,2] have recently appeared. This is a matter, moreover, which is of considerable importance at the present time since certain committees which are now compiling high-temperature creep data for design purposes must select some definite time period for comparing the results from the different laboratories. One of these committees has standardized on tests of 1000-hr. duration and have chosen as a creep rate that occurring between 700 and 1000 hr. The selection of these conditions, however, was based more upon assumptions than upon actual information since very few if any experimental results were available to indicate the influence of the time factor.

The purpose of this paper is to present data from creep tests which have been in progress for several thousand hours and to indicate how the creep characteristics may vary, depending upon the duration of the tests. Also, in order that the complete time-elongation characteristics might be obtained, certain of the stresses were purposely chosen of sufficient magnitude actually to cause rupture at certain periods during the progress of the tests. Results are also presented from metallographic, X-ray and hard-

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[1] P. H. Clark and E. L. Robinson, "An Automatic Creep Test Furnace Guide," *Metals and Alloys*, February, 1935, p. 46.

[2] F. B. Foley, Discussion of Clark and Robinson Article, *Metals and Alloys*, February, 1935, p. 50.

ness examinations of the fractured specimens which were undertaken to determine the influence of the rate of strain on the structural and deformation characteristics.

MATERIALS

Two steels were submitted to the extended-time creep tests, one of which was a plain carbon steel of the S.A.E. 1015 type and the other a low-alloyed chromium-silicon-molybdenum steel. Both were secured from The Timken Steel and Tube Co. and were melted in a commercial electric furnace. The material was heat-treated in the form of 1-in. round bars and the specimens were machined after the heat treatment. Information concerning their chemical composition, heat treatment, Brinell hardness and "inherent" grain size are given in Table I.

Both steels were tested in the same condition of heat treatment, the annealed, and both possessed the same "inherent" grain size. They differed slightly in their hardness with the carbon steel being somewhat the harder. Their chief difference, however, was in their chemical composition.

TABLE I.—CHEMICAL COMPOSITION, HEAT TREATMENT, BRINELL HARDNESS AND GRAIN SIZE OF STEELS.

Designation	Chemical Composition, per cent					Heat Treatment	Brinell Hardness	Grain Size
	Carbon	Manganese	Silicon	Chromium	Molybdenum			
S. A. E. 1015	0.15	0.50	0.23	....	....	Annealed at 1550 F.	134	4 to 5
Cr-Si-Mo	0.07	0.42	0.72	1.25	0.54	Annealed at 1550 F.	123	4 to 5

TEST PROCEDURE

The standard creep-testing apparatus of the University of Michigan, which has been previously described,[1] was used for these tests. The elongation or creep is measured by an optical extensometer system capable of reading to 0.0000028 in. per inch of 2 in. gage length. Standard 0.505-in. diameter specimens were used and the collars of the extensometer system were attached at both ends of the 2-in. gage section. All the testing details were in agreement with the A.S.T.M. Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 34 T).[2]

Five different stresses, ranging from 13,000 to 34,100 lb. per sq. in., were applied to the alloy steel and two, of 4000 and 6000 lb. per sq. in., to the plain carbon steel. Elongation readings were taken daily and the results plotted as time-elongation curves. The slopes (creep rate) of these curves were determined at approximately 500-hr. intervals and the rates thus obtained plotted against the corresponding times. The creep rates, for the various times, were then plotted on logarithmic coordinates with the corresponding loads, and the stresses for definite creep rates (0.01, 0.10 and 1.0 per cent per 1000 hr.) thus computed. In order to show the influence of time on the reported values, these creep values were then plotted against time.

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[1] A. E. White, C. L. Clark and L. Thomassen, "An Apparatus for the Determination of Creep at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 52, No. 27, p. 347 (1930).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1223 (1934); also 1934 Book of A.S.T.M. Tentative Standards, p. 1147.

To facilitate the reproduction of the various figures, the sensitivity of the scale of plotting has in certain cases been greatly reduced. This applies especially to the time-elongation and the time - rate of creep curves. All of the reported values, however, were obtained from the original, and not the reduced, scales. A comparison of the reported values with those computed from the curves as given will indicate the influence of the sensitivity of plotting on the reported creep characteristics.

TABLE II.—TENSILE PROPERTIES AT ROOM AND ELEVATED TEMPERATURES OF CHROMIUM-SILICON-MOLYBDENUM AND PLAIN CARBON S.A.E. 1015 STEELS.

NOTE.—Each value is the average of two tests.

Steel	Temperature, deg. Fahr.	Tensile Strength, lb. per sq. in.	Yield Stress, lb. per sq. in.		Proportional Limit, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
			0.1 per cent Set	0.2 per cent Set			
S.A.E. 1015.............	80	62 425	41 125	42 000	34 500	36.0	67.45
Cr-Si-Mo...............	80	66 500	34 300	35 200	24 000	36.5	72.7
S.A.E. 1015.............	750	58 025	23 875	24 625	13 125	34.25	67.0
Cr-Si-Mo...............	750	71 650	25 450	26 400	18 000	27.0	67.8
S.A.E. 1015.............	900	45 500	22 125	23 500	11 250	37.75	70.6
Cr-Si-Mo...............	900	67 525	24 150	25 600	18 000	25.75	66.35
S.A.E. 1015.............	1000	36 500	18 750	20 125	8 750	42.5	76.9
Cr-Si-Mo...............	1000	57 750	23 500	24 900	15 000	25.5	73.3
S.A.E. 1015.............	1100	27 200	13 300	14 250	5 000	56.75	82.15
Cr-Si-Mo...............	1100	47 500	22 500	23 100	13 500	31.0	82.55
S.A.E. 1015.............	1200	20 000	9 500	10 200	1 875	54.25	89.1
Cr-Si-Mo...............	1200	33 275	14 000	16 000	3 500	36.5	88.2
S.A.E. 1015.............	1300	13 550	6 500	7 375	......	59.5	91.55
Cr-Si-Mo...............	1300	22 775	10 200	11 100	2 250	61.75	94.5
S.A.E. 1015.............	1400	9 025	3 375	3 750	......	69.75	76.9
Cr-Si-Mo...............	1400	13 800	5 950	7 000	1 500	72.25	98.35

The metallographic, X-ray and hardness examinations of the fractured specimens were made at sections close to, and approximately $\frac{3}{4}$ in. from, the fracture.

In order to show more clearly the complete physical properties of these two steels, results are likewise included from tension tests at room and elevated temperatures.

EXPERIMENTAL RESULTS

In addition to the extended-time creep and short-time tension tests, results are included of a metallographic and X-ray examination of the fractured specimens undertaken to determine the influence of time and stress on the resulting structural and deformation characteristics. Hardness values are also reported from the $\frac{3}{4}$-in. section adjacent to the fractures which were made to ascertain the extent to which the strain-hardening characteristics were influenced by these same two factors, that is, time and stress for a given temperature.

Short-Time Tension Tests:

Short-time tension tests were conducted at temperatures over the range of 80 to 1400 F. The results obtained are given in Table II. The yield stress and proportional limit values were determined with the same optical extensometer system used in the creep tests. This reads to 0.0000028 in. per inch of 2-in. gage length. The procedure employed was in accordance with that set forth in the Society's Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 - 34 T).[1]

Check tests were conducted at each temperature and the agreement obtained indicated the steels to be uniform. For example, at room temperature the variations in the tensile strength values of the two chromium-silicon-molybdenum specimens was 500 lb. per sq. in. and for the carbon steel, it was 1500 lb. per sq. in. Differences of this same general magnitude existed over the entire temperature range, not only for the tensile strength values, but for the other properties as well.

Creep Tests:

Results are presented from creep tests at 1000 F. on a plain carbon steel of the S.A.E. 1015 type and on a chromium-silicon-molybdenum steel, both of which were in the annealed condition. Two stresses, of 4000 and 6000 lb. per sq. in., were considered in the case of the carbon steel and five, ranging from 13,000 to 34,100 lb. per sq. in., with the alloy steel. The tests on the plain carbon steel have now been in progress for approximately 8500 hr. and those on alloy steel from 2000 to 8000 hr. The time-elongation curves obtained are given in Figs. 1 and 2. As stated previously, the sensitivity of the elongation scale has been greatly reduced in both figures to facilitate reproduction.

Time Versus Elongation.—Figure 1 gives the results obtained with the carbon steel. It is to be noted that the curve for the lower stress, 4000 lb. per sq. in., is plotted to a scale ten times as sensitive as that used for

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1214 (1934); also 1934 Book of A.S.T.M. Tentative Standards, p. 1138.

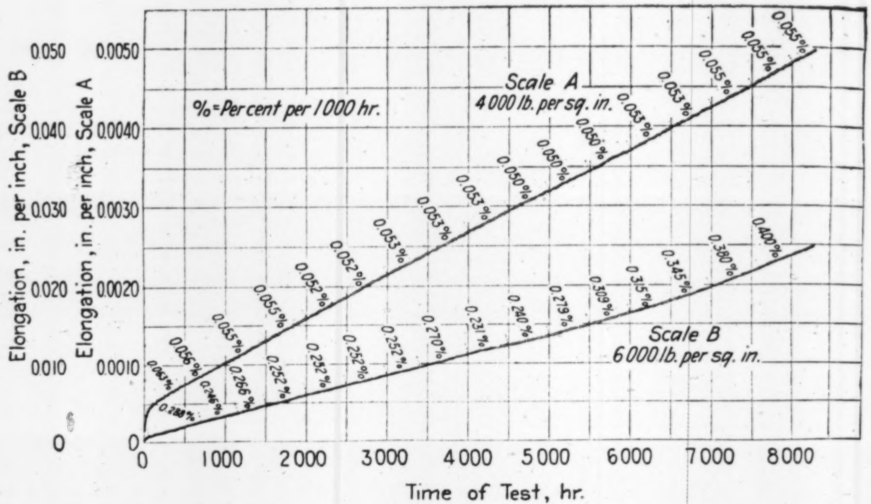


FIG. 1.—Time-Elongation Curves at 1000 F. for S.A.E. 1015 Steel (Electric).

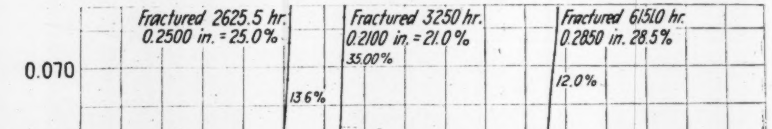
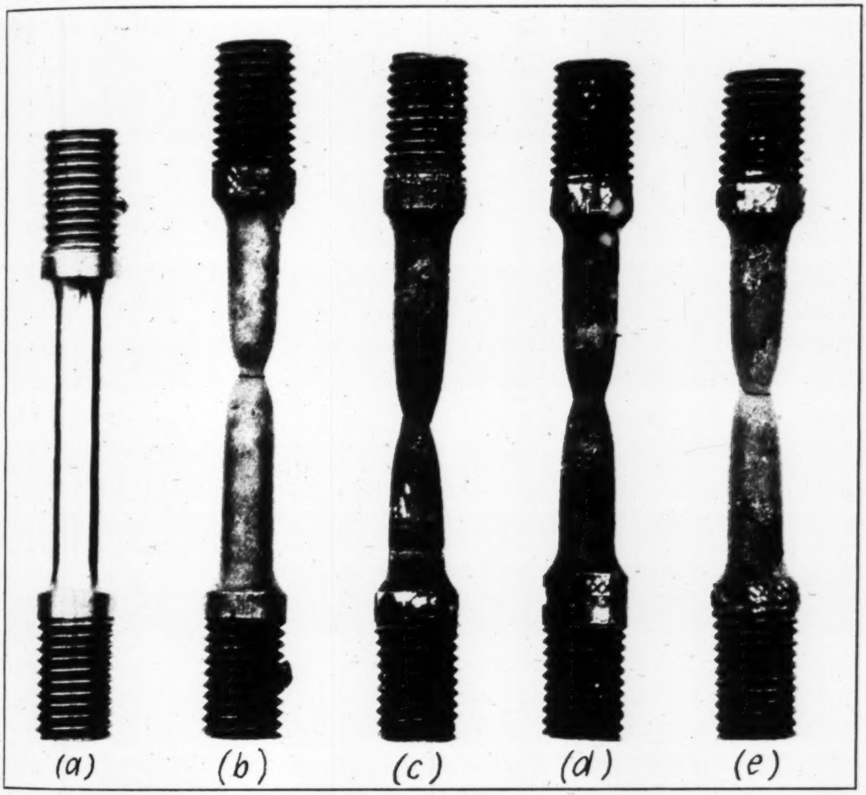


FIG. 2.—Time-Elongation Curves at 1000 F. for Chromium-Silicon-Molybdenum Steel.

the larger stress. The rate of creep (slope) was determined for various time-intervals, which averaged about 500 hr., and the resulting rates, expressed in terms of per cent per 1000 hr., are indicated on the curves. Neither of these specimens has as yet fractured and the tests are still in progress.



(*a*) Original Specimen.
(*b*) Tension Specimen. 1000 F.; elongation in 2 in., 32.5 per cent; reduction of area. 78.6 per cent.
(*c*) Creep Specimen. 1000 F.; 24,600 lb. per sq. in.; 6151 hr. for rupture; elongation in 2 in., 28.5 per cent; reduction of area, 72.8 per cent.
(*d*) Creep Specimen. 1000 F.; 30,000 lb. per sq. in.; 3250 hr. for rupture; elongation in 2 in., 21.0 per cent; reduction of area, 67.9 per cent.
(*e*) Creep Specimen. 1000 F.; 34,000 lb. per sq. in.; 2625 hr. for rupture; elongation in 2 in., 25.0 per cent; reduction of area, 70.3 per cent.

FIG. 3.—Influence of Time on the Resulting Fracture of Chromium-Silicon-Molybdenum Steel at 1000 F.

Corresponding results for the alloy steel are given in Fig. 2. In this case certain of the stresses were purposely chosen of sufficient magnitude to cause fracture. This was done in order that the complete time-elongation curves would be obtained, thus showing the behavior of the material in the so-called third stage of creep (stage of increasing creep rate), as well as in the first and second stages. In order to produce this condition, however, the stresses necessarily were considerably greater than those which would ever be considered for commercial applications. It will be observed that with the stress of 34,100 lb. per sq. in., fracture was obtained in 2625 hr., with 30,000 lb. per sq. in. in 3250 hr., and with 24,600 lb. per sq. in. in 6151 hr. The remaining specimens have not failed and the tests are still being continued. With the stress of 17,500 lb. per sq. in., however, the time-elongation curve appears to be in the third stage, indicating that fracture may eventually occur.

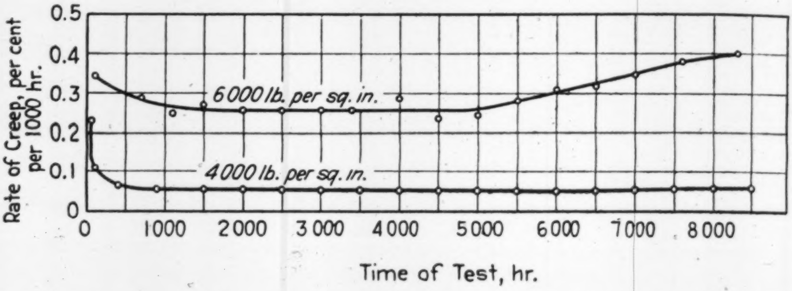


FIG. 4.—Influence of Time of Test on the Observed Creep Rate. Carbon Steel at 1000 F.

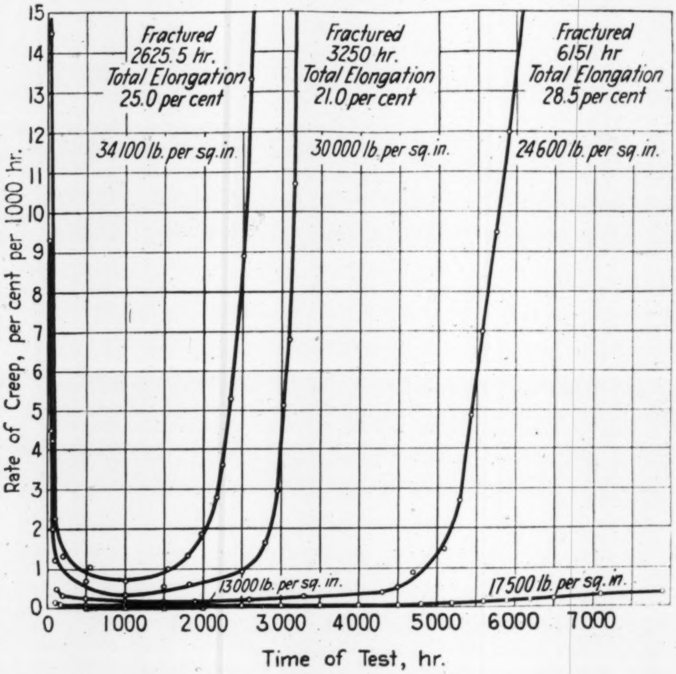


FIG. 5.—Influence of Time of Test on the Observed Creep Rate. Chromium-Silicon-Molybdenum Steel at 1000 F.

Figure 3 shows the fractured specimens. An original specimen, as well as a short-time tension specimen broken at 1000 F., are included for comparative purposes. It will be noted that fracture occurred at approximately the center of the gage section in all cases. There are many points of similarity between the specimens. All of the fractures are of the cup-and-cone type, no marked oxidation has occurred, and the ductility characteristics do not vary greatly. The elongation in 2 in. ranged from 32.5 to 21.0 per cent and the reduction of area from 67.9 to 78.6 per cent. A uniform gradient in these values does not exist. That is, there is no apparent relationship between the time required for rupture and the resulting ductility. The only conclusion which may be drawn is that none of the fractured creep specimens possessed as high a degree of ductility as the short-time tension specimens, but that the degree of observed reduction is not sufficient to cause undue alarm.

The behavior of the chromium-silicon-molybdenum steel, after the so-called third stage is reached, is significant. It is commonly supposed that fracture results soon after this stage is entered. Yet in the case of the 24,600 lb. per sq. in. stress this stage occupied 4000 hr. and the specimen under the stress of 17,500 lb. per sq. in. has already been in this stage for 3500 hr. with the specimen being far from ready for fracture. This fact is important since it not only indicates that considerable swelling, in the case of tubes, will occur before fracture, but also that considerable time may be required for fracture after the swelling does occur.

Tests are now in progress on the carbon steel under stresses greater than 6000 lb. per sq. in. in order that the third-stage characteristics of this material, under different stresses, can be determined. A complete set of curves, as shown for the alloy steel in Fig. 2, will then be available for the carbon steel.

Time Versus Creep Rate.—In order to show the influence of time on the resulting creep rates for the stresses considered, the slopes (creep rates) of the curves of Figs. 1 and 2 are plotted against time in Figs. 4 and 5. In these two figures the creep rates at the various designated times are defined as the rates which would have been reported had the tests been discontinued at that particular time. It should also be emphasized that the creep rates considered are those occurring in the so-called second stage and not for the entire test period. In other words, creep occurring in the first stage was not considered although the time intervals selected for computing creep rates over the earlier portions of the tests were in general sufficiently small to include the larger share of the first stage deformation.

The results from the plain carbon steel, Fig. 4, indicate that under the lower stress, the creep rate decreased rapidly during the first 500 hr. of the test, after which it remained practically constant as the test was extended to 8500 hr. Under the larger stress, the decrease continued for the first

2000 hr., became constant over the period from 2000 to 5000 hr., and then increased for the remainder of the test. There is a possibility that the increase in creep rate which occurred after 5000 hr. is not caused by the steel having entered the third stage of creep but is due to structural changes, such as spheroidization, having decreased the creep resistance. Additional results will indicate which of these two assumptions is correct.

The corresponding results for the chromium-silicon-molybdenum steel are given in Fig. 5. As before, the creep rates decreased during the early portion of the tests, with the decrease being more pronounced, the greater the applied stress. The period over which the decrease occurred, however, varied inversely with the stress. With the two higher stresses, the minimum rate occurred at approximately 1000 hr., with the 24,600 lb. per sq. in. stress, at 2000 hr., and with the 17,500 lb. per sq. in. stress, at 4500 hr. In the tests which have fractured to date a very rapid increase in the creep rate occurred after a rate of 1.0 per cent per 1000 hr. was passed.

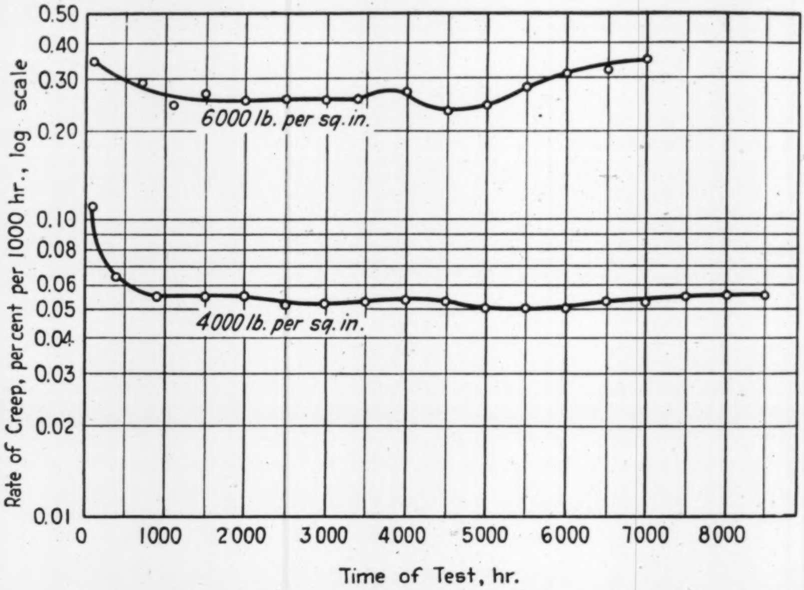


FIG. 6.—Influence of Time of Test on the Observed Creep Rate. Carbon Steel at 1000 F.

There is a question as to whether deformation rates greater than 1.0 per cent per 1000 hr. can be considered as creep. The word creep is usually reserved for slow rates of flow. On this basis it might be assumed that the creep characteristics predominate up to a rate of 1.0 per cent per 1000 hr. and the short-time tensile properties above this rate. The data in Fig. 5 seems to indicate a definite limit for the rate of creep that could be properly designated as creep.

In order more clearly to reveal the data given in Fig. 5 for the alloy steel, especially for the lower stresses, the results are plotted to semi-logarithmic coordinates in Fig. 7, with similar results for the carbon steel being given in Fig. 6. The resulting curves in these figures have been drawn through the actual points rather than reproduced as smooth average curves. This procedure was followed since, especially in the case of the alloy steel, uniform changes appear to occur as the stresses are decreased. For example, in Fig. 7, two points of inflection are obtained to the left of the minimum creep rate under the highest stress, 34,100 lb. per sq. in., and none to the right of this point. With the next highest stress, 30,000 lb. per sq. in., the inflections to the left of the minimum point are much less pronounced and one appears to the right. With the 24,600 and 17,500 lb. per sq. in. stresses no points of inflection appear to the left of the minimum point, but four occur to the right for the 24,600 lb. per sq. in. stress and evidently

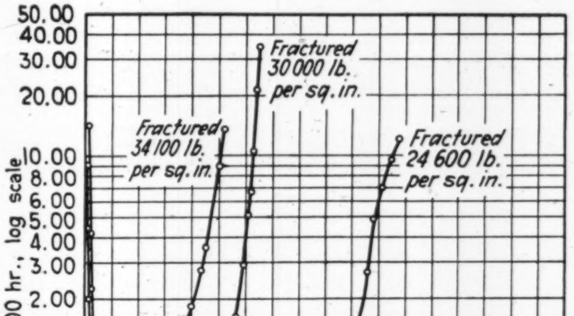


Fig. 7.—Influence of Time of Test on the Observed Creep Rate. Chromium-Silicon-Molybdenum Steel at 1000 F.

several will occur with the 17,500 lb. per sq. in. stress before fracture is obtained. No interpretation is at present offered for these transitions, but as additional data are obtained, an explanation may be possible.

Time Versus Creep Strength.—It is customary to determine the creep strength from time-elongation curves, such as those shown in Figs. 1 and 2, by plotting to logarithmic coordinates the stresses and corresponding creep rates. A straight-line relationship often results and it is thus possible, within certain limits, to determine the stress corresponding to any given creep rate, or the creep rate corresponding to a given stress. Such a procedure was followed in this case and the creep stresses, corresponding to given rates, were determined for each 500-hr. period of the tests. In order to avoid excessive extrapolation, only the stress for 0.10 per cent per 1000 hr. was determined for the carbon steel since it so happened that the creep rates for the two chosen stresses fall on both sides of and fairly near to this value. Likewise with the alloy steel this same value was determined over the entire test period. With this steel, however, the experimental creep rates were such that the stress for 1.0 per cent creep per 1000 hr. could also be obtained in the majority of cases, and also the stress for a creep rate of 0.01 per cent over the early portion of the test.

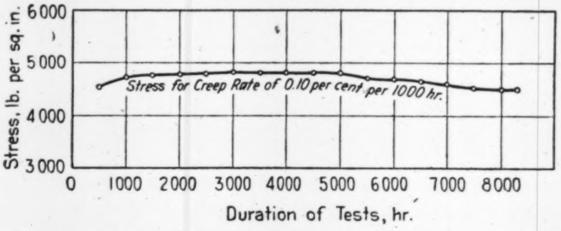


FIG. 8.—Influence of Duration of Tests on Reported Creep Stress. Carbon Steel at 1000 F.

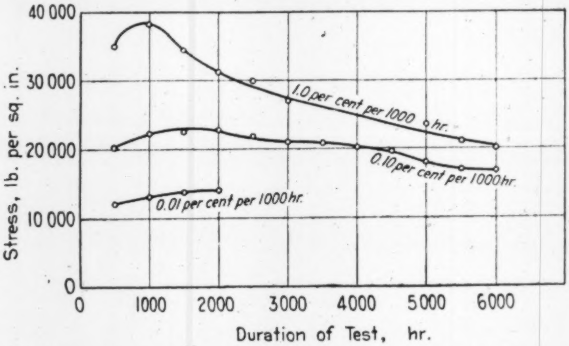


FIG. 9.—Influence of Duration of Tests on Reported Creep Stress. Chromium-Silicon-Molybdenum Steel at 1000 F.

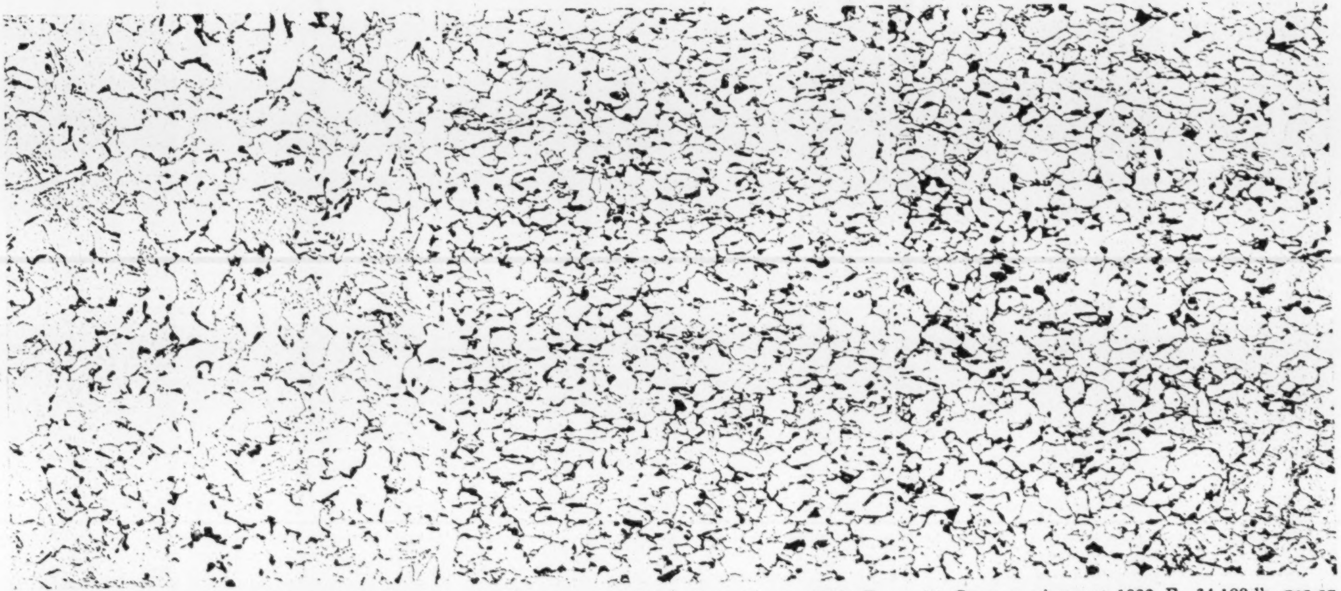
In Figs. 8 and 9 the creep stress for the plain carbon and the chromium-silicon-molybdenum steels, respectively, are plotted against time. As before, the creep stresses at the various designated times are defined as the values which would have been reported had the tests been discontinued at that particular time. First considering the carbon steel, Fig. 8, the creep stress for 0.1 per cent creep per 1000 hr. increased slightly for 1500 hr., then remained constant up to 5000 hr., after which it decreased for the remaining portion of the test. The observed variations in this value are not marked, however, as the minimum value is 4520 and the maximum, 4800 lb. per sq. in. It is also interesting to note that the 500-hr. test gave a lower value than did the tests up to 5000 hr. in length and further that this value agrees with that obtained after 8000 hr. In other words, for this particular steel at least, the 500-hr. test is a more conservative indication of the true behavior under prolonged service at 1000 F. than are tests up to 5000 hr. in duration.

The corresponding variations in the creep strength of the chromium-silicon-molybdenum steel with time are given in Fig. 9. In this case stresses are included for creep rates of 0.1 and 1.0 per cent per 1000 hr. over the entire period of the tests, and also for a creep rate of 0.01 per cent for the first 2000 hr. of the tests. As with the plain carbon steel, the stresses for the designated creep rates first increased as the time of testing was extended above 500 hr. In the case of the stress for the highest creep rate, the maximum occurred at 1000 hr., for the medium creep rate at 1500 hr., and for the lowest creep rate the stress value is still increasing at 2000 hr. Time exerts its greatest influence on the stress required for the maximum creep rate as the values range from 38,500 to 20,000 lb. per sq. in. The corresponding range in the stresses for the medium creep rate is from 22,500 to 16,700 lb. per sq. in. It is again to be noted that the 500-hr. test is a more conservative indication of the true creep strength than is one of 1000 hr. In fact, in the case of the stresses required for the maximum creep rate the 500 and 1500-hr. tests yield approximately the same values. Likewise, with the stresses required to produce the medium creep rate the same is true of the 500 and 4000-hr. tests.

Metallographic Examination:

In order to determine the influence of stress and time at 1000 F. on the resulting metallographic structures, the fractured creep specimens, as well as the original material and the short-time tension specimens broken at 1000 F., were examined. Sections were selected at the fracture and at a distance of approximately $\frac{3}{4}$ in. from it, and magnifications of 100 and 1000 diameters were used. In order to avoid duplication, only typical structures are given in Figs. 10, 11 and 12.

The apparent grain size of the creep and tension specimens, Figs. 10 and 11, is considerably smaller than that of the original material, indicating that at the section $\frac{3}{4}$ in. from the fracture the time at temperature

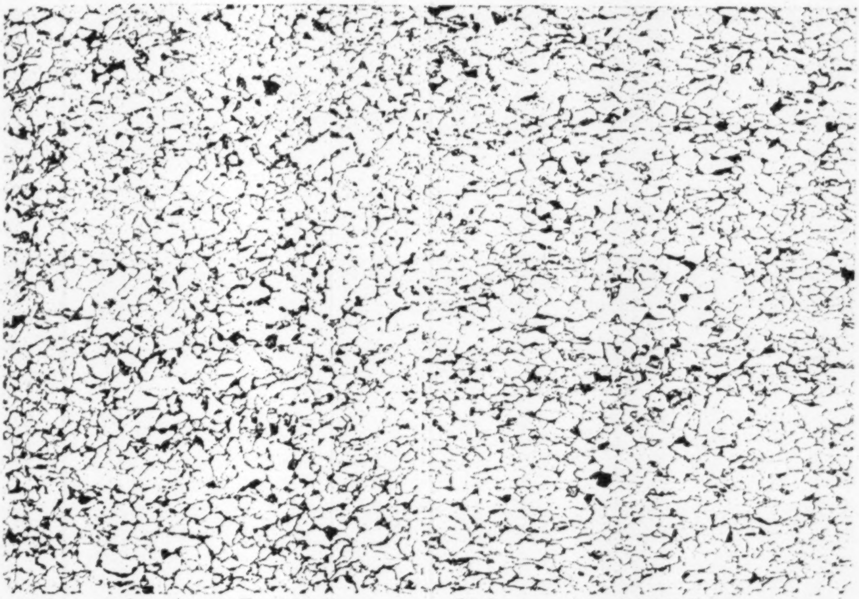


(*a*) Original structure, longitudinal section. Note fairly large, equi-axed grains and absence of definitely laminated pearlite.

(*b*) Short-time tension specimen at 1000 F. Longitudinal section $\frac{3}{4}$ in. from fracture. Note grain refinement and distortion.

(*c*) Creep specimen at 1000 F., 34,100-lb. per sq. in. stress. Longitudinal section $\frac{3}{4}$ in. from fracture. Note that grain is larger than in (*b*) and smaller than in (*a*). Grains are fairly equi-axed.

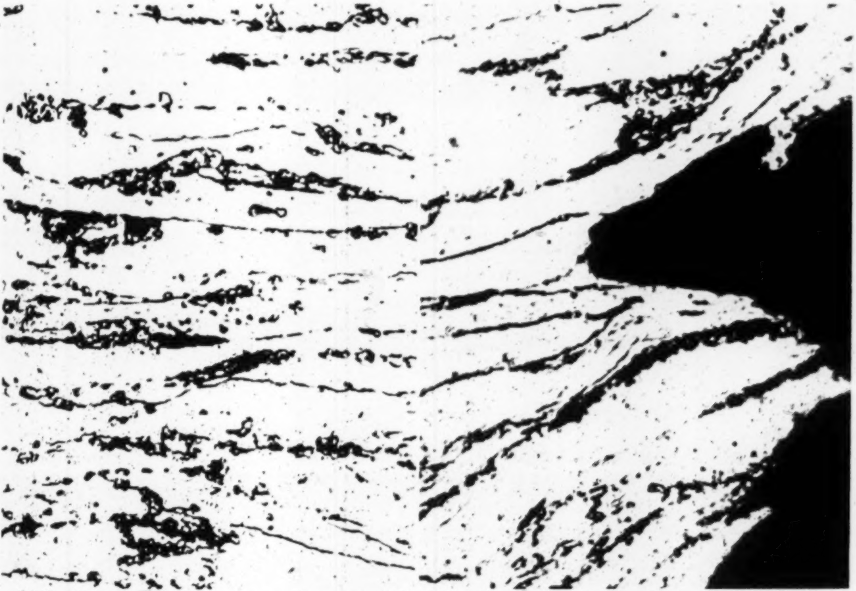
FIG. 10.—Microstructure of Chromium-Silicon-Molybdenum Steel Before and After Short-Time Tension and Creep Tests at 1000 F. (× 100).



(*a*) Creep specimen at 1000 F., 30,000-lb. per sq. in. stress. Longitudinal section ¾ in. from fracture. Note non-uniform grain size but with average size smaller than in Fig. 10 (*c*).

(*b*) Creep specimen at 1000 F., 24,600-lb. per sq. in. stress. Longitudinal section ¾ in. from fracture. Note fairly uniform grain of smaller size than in (*a*).

FIG. 11.—Microstructure of Chromium-Silicon-Molybdenum Steel After Creep Tests at 1000 F. (× 100).

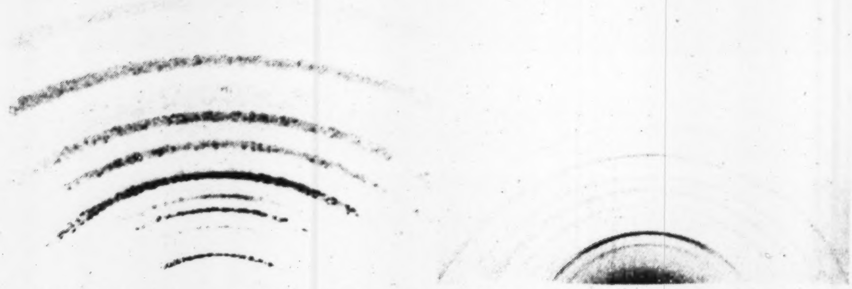


(*a*) Section adjacent to fracture. Note marked elongation of both ferritic and pearlitic grains.

(*b*) Section at fracture. Note marked elongation of the grains and lack of indication as to whether fracture is inter- or trans-crystalline.

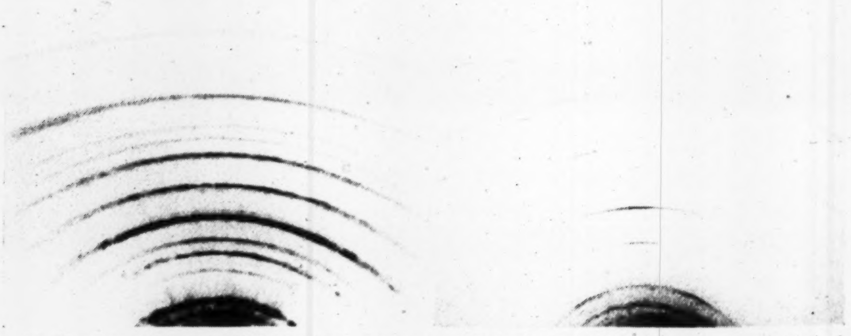
FIG. 12.—Microstructure of Chromium-Silicon-Molybdenum Steel in Reduced Section of Creep Specimen (× 1000).

and deformation conditions were such that refinement of the grain occurred. It is also to be noted that the grain size of the creep specimen becomes smaller and more uniform as the time for fracture is increased, or in other words, as the applied stress is decreased. The grain size of the short-time tension specimens is between that of the creep specimens subjected to stresses of 34,100 and 30,000 lb. per sq. in., indicating that stress (deformation) as well as time is a controlling factor on the resulting grain size.



(*a*) Original structure. Note comparative freedom from strain.

(*b*) Short-time tension specimen. Pulled at 1000 F. Note indications of severe strain and crystal fragmentation.



(*c*) Creep specimen at 1000 F., 24,600 lb. per sq. in. Note indications of strain but absence of crystal fragmentation.

(*d*) Section at point of maximum reduction and typical of both creep and tension specimens. Note presence of severe strain, crystal fragmentation and preferred orientation of the crystals.

FIG. 13.—Typical Films Resulting from an X-ray Examination of Fractured Tension and Creep Specimens.

The grains of the short-time tension specimens are also more distorted than those of the creep specimens. Since the sections examined were all at the same distance from the actual fracture, this indicates a more pronounced stress concentration in the short-time tension specimen.

The structure of the original steel was partially spheroidized and the fractured specimens did not show appreciable differences in the degree of spheroidization. Conclusive evidence is not, therefore, available as to whether spheroidization will occur at 1000 F. under the given test conditions. It is known, however, that the carbon steel does have its pearlite in a laminated condition in the original state. An examination of these specimens upon the completion of the tests will offer further information in this respect.

As would be expected the grains in the immediate vicinity of the fracture are in a severely strained and elongated state, Fig. 12 (*a*). No apparent differences could be detected between the four specimens and this is without doubt due to the fact that this particular section represents conditions occurring during the very last portion of the tests, conditions which should be approximately the same regardless of the total time required for rupture. The actual fracture, Fig. 12 (*b*), was also examined to determine whether it were intercrystalline or transcrystalline in character. Because of the nature of the fracture, however, definite information could not be obtained.

X-ray Examination:

Various theories have been advanced as to the possible mechanism of creep and it was felt that an X-ray examination of the fractured specimens might shed additional light on this subject. Examinations were by surface reflection of the X-rays, produced in a tube equipped with a mobybdenum target, from the specimen both at the fracture and at a section $\frac{3}{4}$ in. from it. The X-rays were passed both perpendicular and parallel to the axes of the specimens and both the outer surface and mid-section were considered. A number of X-ray films were obtained showing the same general results but only four typical ones are included in Fig. 13.

Two of the films which are reproduced were taken at a section approximately $\frac{3}{4}$ in. from the fracture, for it was found, as in the case of the metallographic examination, that no appreciable differences existed in the various specimens at a section immediately adjacent to the fracture as shown in Fig. 13 (*d*). As stated previously, it is believed that this is due to the fact that the conditions existing in this section are produced by the behavior during the last portion of the test, that is, after considerable necking has occurred, and that this behavior is approximately the same in all cases.

The X-ray films indicate the original steel to be relatively free from strain as shown in Fig. 13(*a*). It is at least comparable in this respect to the commercially annealed steels usually encountered. The creep specimens, Fig. 13(*c*), contained more strain than the original structure, but no appreciable fragmentation of the grains had been produced. The short-time tension specimens, Fig. 13(*b*), were considerably strained and the grains were fragmented.

It is evident, therefore, that a difference does exist between the short-time tension and creep specimens in that the creep specimen shows strain without fragmentation, while the tension specimen shows both fragmentation and strain. Since the total elongation or deformation was approximately the same, there are two possible assumptions to account for the absence of fragmentation in the creep specimen. Either the process of deformation was such that fragmentation did not occur or else the time held at temperature was sufficient to remove the effects of this fragmentation. The present results do not, therefore, either prove or disprove the various theories which have been advanced for the mechanism of creep and additional work will be required before definite conclusions may be reached.

TABLE III.—VICKERS HARDNESS TESTS ON FRACTURED TENSION AND CREEP SPECIMENS.

Position on Specimen	Original	Tension Specimen	Creep Specimen		
			34,100 lb.	30,000 lb.	24,600 lb.
¾ in. from fracture	138	187	157	155	157
		183	160	155	157
		191	160	160	162
		194	162	165	168
		194	171	171	171
		210	180	174	168
At fracture	...	214	183	177	168

Hardness Tests:

In a further attempt to determine differences in straining effects with time, hardness tests were conducted on each of the fractured specimens over the ¾-in. section adjacent to the fracture. Various methods were tried but the Vickers was found to yield the most satisfactory results. Table III gives the findings obtained.

All of the fractured specimens possessed a greater hardness, over the area considered, than the original steel, indicating that sufficient strain-hardening had occurred to produce a measurable effect. The tension specimen was harder than any of the creep specimens and the maximum observed hardness in the creep specimens decreased as the time necessary for rupture increased. These observed differences can again be accounted for either by a difference in the deformation characteristics or by the annealing effect of prolonged time at 1000 F.

CONCLUSIONS

Extended-time creep tests conducted at 1000 F. on plain carbon steel and chromium-silicon-molybdenum steel, as well as metallographic, X-ray and hardness examinations of the fractured specimens allow the following conclusions:

1. In all the creep tests, the rate of creep decreases and thus the creep strength increases during the earlier periods of the test and the magnitude of the changes, for the given temperature, depends both upon the steel and the stress being considered. If the stress is sufficiently great, the creep rate will increase after a certain time and the time required for this change varies inversely with the stress.

2. The reported creep stresses will, therefore, vary depending upon the length of time the tests are conducted. In the case of the carbon steel at 1000 F. the amount of variation was not excessive as the values for a creep rate of 0.10 per cent per 1000 hr. ranged only from 4500 to 4800 lb. per sq. in. as the testing time was extended for 8500 hr. The changes were, however, more pronounced with the alloy steel due to the considerably greater stresses employed. The reported stresses for the creep rate previously mentioned ranged from 16,700 to 22,700 lb. per sq. in. with even a greater difference, 20,000 to 38,500 lb. per sq. in., existing for a creep rate of 1.0 per cent per 1000 hr. This would indicate that the observed differences with time may be proportional to the magnitude of the creep rate being considered.

3. The ability of relatively short-time creep tests (500 to 2000 hr.) to prophesy the behavior under more extended time periods varies, for the given temperature, with the steel considered. With a steel in which the net strain-hardening effect is slight, such as the plain carbon steel in this case, the time factor is not of great importance and a test of 500-hr. duration gives a good indication of the load-carrying ability for periods of several thousand hours. For other cases in which the resulting strain-hardening effect is more pronounced, such as with the alloy steel, the reported values vary considerably over the period of 500 to 2000 hr. In both of the cases considered, the 500-hr. test gives lower creep strength values than does the 1000-hr. test and also values which correspond more closely with those obtained from tests of several thousand hours duration. The results would appear to indicate, therefore, that at least in the two cases considered, the 500-hr. test is a more conservative criterion than the 1000-hr. test.

4. The assumption ordinarily made that a creep rate of 0.10 per cent per 1000 hr. represents a creep rate of 1.0 per cent per 10,000 hr is in consequence open to question, at least for some steels. Indications are that for the plain carbon steel this hypothesis may be approximately true but for

the chromium-silicon-molybdenum steel this relationship was not found. In fact, the stress giving this creep rate at the end of 1000 hr. produces, after 5000 hr., a creep rate of 1.0 per cent per 1000 hr.

5. The fact that a specimen enters the third stage of creep does not necessarily imply that it will fail in a short time. This is well illustrated by the behavior of the alloy steel under stresses of 24,600 and 17,500 lb. per sq. in. In the first case, the third stage required 4000 hr. before rupture and with the lower stress, considerably greater time will apparently be necessary.

6. The time required for rupture influences the resulting ductility values but not in a uniform manner. The fractured creep specimens of the chromium-silicon-molybdenum steel all possessed a lower ductility than that obtained from the short-time tension test at the same temperature, but the variations in the values of the creep specimens were not proportional to the time required for fracture. Neither were the changes of sufficient magnitude to cause undue alarm.

7. On the basis of the metallographic examination, structural changes were produced during both the tension and creep tests. All of the fractured specimens possessed a smaller grain size than the original steel and the grain size of the creep specimens became smaller, the greater the time required for rupture. Results also indicate that no appreciable spheroidization occurred during the tests. The findings do not permit too definite conclusions in this respect, however, since the original structure was partially spheroidized.

8. The X-ray examination revealed a difference in the apparent deformation characteristics. The original steel was relatively strain free, the creep specimens were strained, while the short-time tension specimen was not only strained but considerable crystal fragmentation had occurred.

9. A greater degree of strain-hardening was evident in the short-time tension specimens than in the creep specimens. The maximum hardness of the creep specimen decreased as the time required for rupture was increased.

10. The changes observed from the metallographic, X-ray and hardness examinations may be accounted for either on the assumption of a difference in the mechanism of deformation in the short-time tension and creep specimens, or that, in the case of the creep specimens, the time held at temperature was sufficient to remove certain of the effects of the deformation.

Acknowledgments:

The authors desire to express their appreciation to The Timken Steel and Tube Co. for their financial assistance which has made these investigations possible and for their permission to publish the findings. Particularly do they wish to express their gratitude to F. J. Griffiths, H. H. Timken, Jr., W. H. Wiewel and K. B. Bowman.

DISCUSSION

Mr. P. G. McVetty[1] (*presented in written form*).—The authors of this paper have made a valuable contribution to our knowledge of creep phenomena by demonstrating the important influence of time upon creep rates. A considerable quantity of creep data has been published in the past in which a definite creep rate is given for each combination of stress and temperature. In using such data, it is inferred that one may expect creep at this constant rate over a long period of time. The data here given show the necessity of studying the original creep data carefully before making any assumption as to the probable constancy of creep rate.

Any creep curve which has a point of inflection as shown by the four curves in Fig. 2 of the paper is characterized by a change from a decreasing to an increasing creep rate at this point. Differentiation of these creep curves gives creep rate curves of the type shown in Fig. 5. The effect of stress is clearly shown in this figure. The change of creep rate which is rapid at high stress, decreases as the stress is reduced until the time at or near the minimum rate becomes extremely long for the relatively low stresses commonly used in design for high temperature service.

A convenient method of predicting the effect of long periods of time on creep rate consists in substituting total creep for time in plotting the curves of Fig. 5. At low stresses and low creep rates a very long time is necessary to produce the same total creep as is obtained in a short time at high stress. Since the curves cannot cross, the low stress curves may be extrapolated with fair accuracy. A relatively short extrapolation in terms of total creep is equivalent to a considerable extrapolation in time. This expedient may be applied to any family of relatively high-stress creep curves to show the probable time effects at the more useful lower stresses.

In addition to the increased time at or near the minimum creep rate as the stress is decreased, it is important to note the great increase in time required to reach the inflection point. The assumption that the rate of increase of creep rate after is about the same as the rate of decrease before the inflection is shown by Fig. 5 to be reasonably accurate. It is difficult to justify extrapolation of creep curves to times representing service life without analysis of the effect of time on creep rate.

In the use of creep data as a basis for the assignment of working stresses, there is some objection to the logarithmic plot of stress *versus* creep rate. This objection arises solely from the possibility of misinterpreting such curves. The designer may be limited to a total plastic deformation of 0.2 per cent in 20 yr. If he assumes a constant creep rate from the start, this reduces to 0.0114 per cent per 10,000 hr. A stress corresponding to this creep rate taken from the logarithmic stress - creep rate curve will be too high because the more rapid creep during the first stage is neglected.[1] While consideration of first stage creep does not enter this discussion of time effects, it does have an important bearing on the practical application of creep data to design problems.

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[1] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

The authors mention a possibility of structural changes during the test. In addition to the known decrease in creep resistance which accompanies spheroidization of cementite, it is possible also that a volume change may increase or decrease the measured value of creep. When aging phenomena occur during a creep test, either shrinkage or extension may be found in the absence of stress. It is difficult to separate the effect of stress on this change since stress also produces the normal form of creep. The structural changes found by metallographic examination might be expected to cause appreciable changes in creep rate between the start and end of the test. These factors must be considered in any extrapolation of creep tests.

It is of interest to compare the X-ray spectograms with Bailey's concept of creep as the net effect of strain-hardening and annealing. The indications of strain in Fig. 13 (*c*) result from a plastic deformation of 8 per cent or more, as shown in Fig. 2. While most of this deformation occurred during the last 1000 hr. of the test, this time of exposure to a temperature of 1000 F. has not been sufficient to remove all of the strain hardening. This is shown also by the increase in Vickers hardness from 138 to 164.

MR. E. L. ROBINSON[2] (*presented in written form*).—The results presented in this paper are very significant and illustrate strikingly the difference in performance between an alloy steel and plain carbon steel. The duration of these tests is almost a year and a great many more year-long tests ought to be made. Since the test is made both at constant stress and at constant temperature, the only primary variable being time, it is possible to examine alterations of behavior in view of the elapsed time.

These tests have been made purposely at excessive loadings and carried to excessive strains. This brings out the difference between the fairly steady rate in the case of the carbon steel as contrasted with the distinctly "S"-shaped curves of the alloy steel.

There are a great number of applications in which the total strain in any practical application may not exceed the elastic strain. All cases of bolting and shrink fit assemblies are of this nature. The total allowable creep before the assembled device becomes inoperative is not more than about 0.25 per cent. If consideration of creep results is limited to total values not exceeding 0.25 per cent, it will be seen that the region of steady flow is a much smaller fraction of the whole and that the initial effects become of very much greater relative importance. If tests are conducted in which the loading is relieved to correspond with the relaxation of elastic stress that occurs due to creep in the types of application referred to, it is not so easy to analyze changes of behavior with reference to time. There are two primary variables, time and stress and at least one primary resultant, the total creep. It is customary to compare strengths on the basis of creep rates, that is, instantaneous speed at any time. (While there has been in the past, some confusion as to constancy of rate for 1000 or 10,000 or 100,000 hr., there is a tendency at present to clear up this uncertainty by expressing rates in terms of millionths inches per inch per hour, thus, frankly recognizing the tendency of rates to change under the influence of time.)

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[1] See Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 35 T), Appendix II, *Proceedings*, Am. Soc. Testing Mats., Vol 35, Part I, p. 1291 (1935); also 1935 Book of A.S.T.M. Tentative Standards, p. 1415.

[2] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

When time is the only primary variable, it is easy to speak of changes in the relation between stress and resulting rate as due to the influence of time. When both stress and time are primary variables, it is not so easy to see what is the significant influence. There is good reason to believe that the total creep is a good index of changing relationship between strength and rate, especially in the early stages of flow. Total creep is a sort of measure of the degree of strain hardening which has occurred. In a sense, it is an integrated record of the combined influence of time and stress incurred to date.

The writer would like to suggest that in addition to a consideration of time as an influence on creep strength of materials, a consideration of total creep is also valuable, especially when comparisons are to be made between tests conducted at constant stress and relaxation or step-down tests. Both types of application are important and both types of test are described in the literature.

MR. H. F. MOORE.[1]—This very interesting paper has raised two questions in my mind. The first concerns the procedure to be used in determining creep rate. A typical creep-time diagram shows three stages, (1) a short period in which the creep rate diminishes, (2) a long period in which the creep rate is nearly constant, and (3) a final period, leading eventually to fracture in which the creep rate increases. Should the third period be considered in determining creep rate? A dogmatic answer to this question is hardly possible at this time, but it is always desirable that in reporting a determination of creep rate it be clearly stated whether this third stage of increasing rate is considered in the determination.

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[1] **Professor of Engineering Materials, University of Illinois, Urbana, Ill.**

The second question concerns the use of the term "fragmentation" as applied to a crystalline grain of metal. Does this term mean an actual fracture of the grain, or is it used to denote a general, and rather slight, disarrangement of the orientation of adjacent crystallites within a crystalline grain?

MR. J. W. BOLTON.[1]—This paper makes it quite apparent that, especially for the higher rate of creep, it is necessary to go to quite long-time tests to evaluate the third stage of creep. Reference to Fig. 2 of my paper[2] and to the upper and lower curves of the figure presented by Mr. Maack in discussion, will show some interesting relationships with the results given in the present paper, irrespective of whether the alloys be steel or non-ferrous. I refer to the relationship of the stress and rate of creep to the behavior of the alloy, as to whether it will go from the second ultimately into the third stage. The authors disclaim the possibility of a general relationship in the behavior of various alloys. However, the discussion of the two papers indicates that there are some characteristic relationships between a number of seemingly dissimilar metals.

MR. D. M. MCCUTCHEON.[3]—I should like to say a few words in connection with the X-ray work carried out in this creep investigation. A number of diffraction tests have been made at the University of Michigan by interested students in addition to the work reported in this paper. In every case the X-ray work was carried out after the creep test had been finished and while some information was gained it was felt by the writer that a great deal more could be learned by carrying out the X-ray work during the creep test. This necessitates overcoming some very serious obstacles in securing a proper set-up and a decision as to the correct X-ray technique. In any case a superficial application is not desirable as the results desired are a clearer insight into the fundamental problem of creep.

One apparently very fruitful and so far quite neglected method of investigation involves the use of the so-called Gerlach type[4] of doublet resolution that is obtained when monochromatic X-rays are diffracted from fairly large-grained metals such as the steels used in this investigation. Considerable work has been done by the writer in determining the correct heat treatment technique necessary to secure nearly perfect resolution of the Gerlach doublets for low and medium carbon steels. Figure 13(*a*) of the paper represents a good commercial anneal but had the same sample been given a controlled laboratory anneal, the individual spots on the pattern would have been very sharp, allowing the reaction of each individual crystal to be studied during the stressing period. While it may be argued that in higher carbon steels this results in a structure somewhat different than that used commercially, it offers an excellent starting material for an X-ray investigation into the fundamentals of creep.

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[1] Chief Chemist and Metallurgist, The Lunkenheimer Co., Cincinnati, Ohio.
[2] J. W. Bolton, "Some Tests on Tin Bronzes at Elevated Temperatures," see p. 204.
[3] Metallurgist, Main Laboratory, Ford Motor Co., Detroit, Mich.
[4] K. Becker, *Zeitschrift für Physik*, Vol. 42, p. 226 (1927).

The authors have contributed generously to our present knowledge of creep phenomena and it is hoped that a supplementary method may be developed which will be able to definitely check the theories advanced by the authors in their work.

MR. R. L. WILSON.[1]—Mr. McVetty's remarks are of particular interest from the standpoint of establishing suitable working stresses for high-temperature design. The present paper is in a sense a progress report on a program of testing that will be continued for a longer period of time. In the circumstances we shall be able to try the method of extrapolating the curves of total creep *versus* rate of creep as proposed by Mr. McVetty. The value of data giving total creep for extended times is recognized as being especially important in the design of machinery intended for operation at elevated temperatures. In other researches we have studied the microstructural changes in steel in relation to creep and know that spheroidization of cementite is associated with loss of creep strength. It would not be unreasonable to expect that volume changes should accompany the spheroidization of carbide although our long-time creep tests may be considered as indicating the net effect upon the material of simultaneous spheroidization, strain-hardening, and annealing in the course of the tests.

Referring to Mr. Robinson's communication, we are quite pleased indeed that he has called attention to the possibility of extracting more information of some use from the data that we have already gathered in connection with these tests. From the load-extension curves for increasing time, we have the elongation in the initial stages of loading that would provide the information Mr. Robinson would like to have. However, the general conclusions would not be altered; an extra set of facts would simply be obtained by another interpretation of the data. We have set out to indicate the effect of steady stress at constant temperature upon the general characteristics of the steel for different periods of time. This of course incorporates such changes as might occur in the initial stages of extension as a result of the loading. We think Mr. Robinson's point is well taken because the additional data would be useful in a large field of applications, as in turbine work where running clearances are quite small, in close fit bolting, and so on.

With regard to the questions raised by Mr. Moore, we agree that it is quite essential to indicate the stage of creep for which creep strengths are reported. It has been customary with the authors to base creep strength upon the second stage of creep, which is the stage where there is steady creep for a long period of time. This avoids both the changing creep rate that occurs on initial loading and also the later stages of creep where the test specimen may finally rupture. Concerning the interpretation to be placed on the "fragmentation" of crystals as resulting from either rapid deformation as in the short-time test, or a creep test having a fairly fast rate of creep, we prefer to think that this fragmentation consists in an arrangement of crystalline substances that is not orderly. There certainly cannot be any great dislocation in the structure, but some disregistry must exist.

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[1] Metallurgical Engineer, The Timken Steel and Tube Co., Canton, Ohio.

Mr. Bolton has drawn attention to the interesting similarity between the creep curves of a valve bronze and the chromium-silicon-molybdenum alloy steel described in this paper. Creep studies of still other materials may show this behavior is common to all metals and alloys. Further tests on plain carbon steel conducted recently by the authors indicate that the creep characteristics of a simple steel are much the same as for the chromium-silicon-molybdenum type.

We appreciate Mr. McCutcheon's remarks on the application of the X-ray as a research tool in the study of creep specimens. Admittedly it would be desirable to undertake the task of making an X-ray study of specimens during the creep test although we could not say whether that would be possible at the present time. The difficulties seem great.

MAKING AND TESTING SINGLE CRYSTALS OF LEAD

By B. B. Betty[1]

Synopsis

The creep characteristics have been determined for specimens composed of only one crystal to eliminate the effect of movement at the grain boundaries, which, together with the deformation of the grains themselves, appear to determine the creep in polycrystalline lead. The method of casting the crystals, including the temperature and temperature gradient control necessary, is described. An inexpensive, rapid and sufficiently accurate method of determining the crystallographic orientation of the crystals is presented, which obviates the necessity of the laborious and time-consuming X-ray method. Results of creep tests of three specimens are given. Planes of slip are identified with definite crystallographic planes known as octahedral planes.

This paper describes the technique and a few preliminary results of a study of the creep of single crystals of lead which was undertaken as a part of the much larger investigation of the creep properties of cable sheathing on which the author is engaged. In making a metallographic study of the mechanism of creep in lead sheathing it was revealed that considerable movement occurred at grain boundaries.[2] It is inconceivable that this can take place without some deformation of the grains.

By testing single crystals the effects of the boundaries are eliminated and some information gained as to the relative amount of creep strain contributed by distortion of the grains.

Method of Making Single-Crystal Specimens

The Bridgman method[3] was used to cast the crystals. These were of rectangular cross-section $\frac{1}{4}$ in. wide by $\frac{1}{8}$ in. thick at the reduced section and were cast to shape so that it was not necessary to do any machining or cutting on that part of the castings used for the test specimens. Figure 1 shows the mold and the arrangement for heating it. The mold was made of duralumin to minimize sticking of the casting to it and was of such design that it could be taken apart for the removal of the specimen.

When casting a specimen, commercially pure lead was melted in a ladle and poured into the mold which had been preheated to the temperature of the molten metal. The mold then was heated well above the melting point to make sure that every particle within the mold was liquid. This step supposedly eliminated many possible nuclei which might act as centers of crystallization when freezing occurred. Next, a steep temperature gradient was established along the mold by making the blast flames impinge upon the top of the mold and by raising a vessel of water as shown in the figure until the extended flanges of the side plates were immersed about ½ in. While maintaining this gradient all temperatures were lowered by turning down the blast flames a little at a time. Freezing began at the pointed lower end of the specimen where several crystals usually started.

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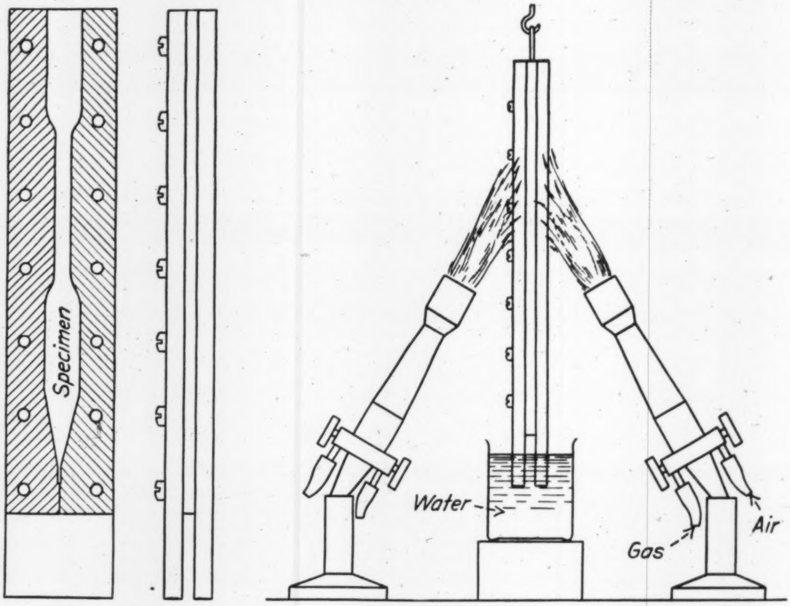
[1] Research Assistant in Engineering Materials, Engineering Experiment Station, University of Illinois, Urbana, Ill.

[2] H. F. Moore, B. B. Betty and C. W. Dollins, "The Creep and Fracture of Lead and Lead Alloys," *Bulletin 272*, Engineering Experiment Station, University of Illinois, February 5, 1935.

[3] P. W. Bridgman, "Certain Physical Properties of Single Crystals of W, Sb, Bi, Te, Cd, Zn and Sn," *Proceedings*, Am. Academy Arts and Sciences, Vol. 60, p. 305 (1925).



(a) Mold (b) General Arrangement

Fig. 1.—Apparatus and Mold for Making Single-Crystal Specimens of Lead.

That crystal which was so oriented that it had the fastest rate of growth along the axis of the specimen, had a tendency to choke the others out. The purpose of the temperature gradient was not only to control the direction in which freezing took place and to conduct away the heat of fusion, but also to reduce the depth of that layer in the melt immediately ahead of the front of solidification in which nuclei might form. In this way the gradient increased the probability of success in obtaining a specimen composed of a single crystal throughout the reduced section.

The shrinkage of the metal as observed at the top of the mold was an indication of the progress of solidification. When the freezing was complete, heat was turned off and the mold allowed to cool in air to 125 F. before the specimen was removed.

The surface of the new casting was quite sensitive to an etchant. One or two seconds in a solution composed of four parts nitric acid (specific gravity 1.72), three parts glacial acetic acid and sixteen parts water, showed whether or not the casting was composed of only one crystal. Different crystals have different directional reflecting power for light and consequently one appears brighter than the other.

DETERMINATION OF CRYSTALLOGRAPHIC ORIENTATION

The preferred directional reflection of light from myriads of tiny facets on the undisturbed surface of a crystal was taken advantage of by P. W. Bridgman[1] in devising a method for determining the orientation of the crystal lattice with respect to its external features. This method has been slightly modified by the author.

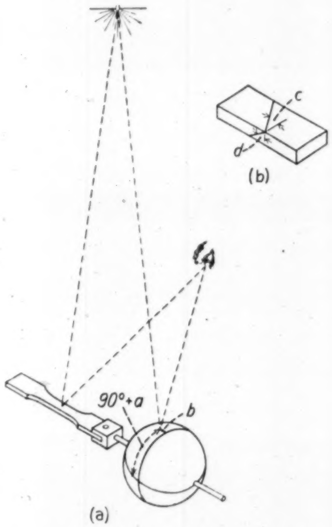


FIG. 2.—Device for Determining Crystallographic Orientation of Specimen.

A polished ball $3\frac{1}{2}$ in. in diameter of almost perfect spherical shape had a small rod passed centrally through it on one end of which was a brass grip. The specimen to be examined was fastened in this grip so that its longitudinal axis lay along the axis of the rod extended, as shown in Fig. 2. Two great circles were drawn on the sphere, one perpendicular to the axis and was designated the zero latitude circle, the other circle lay in a plane parallel to the large face of the specimen and passed through the axis. The latter was designated the 90-270 deg. longitude circle. This having been done, the assemblage was taken to a darkened room with a single light on the ceiling and held at arm's length toward the floor immediately under the lamp. It was rotated until a flash of light, constituting a regular reflection from the facets on the surface, was observed. While being held in this position the point on the polished sphere at which the image of the lamp could be seen was marked. This is the point of tangency on the sphere of a plane which is approximately parallel to the tiny facets reflecting the light from the crystal. By further rotating and spotting points on the sphere it was found that eight reflections were possible. The number and relative location of these eight points (four pairs) show that they correspond to the four sets of octahedral planes of the space lattice. These planes make equal angles with the three rectangular axes of the cubic lattice. By taking the average of the latitudes (in degrees) for each pair of spots and the average for the longitudes, taking due account of signs of course, the error due to angularity of the light balanced out and an accuracy of ±2 deg. was obtained. Once the spots are marked on the sphere, it is only necessary to measure their longitude and latitude with a protractor to establish the orientation of the space lattice.

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[1] *Loc. cit.*

If, on the other hand, the relation between planes of slip in the specimen and the planes of the space lattice is desired to be known some additional calculations are necessary. So far, in the short-time tension tests of metallic single crystals of the face-centered cubic lattice, the only planes on which slip has been observed are the octahedral planes.[1] Since lead has this lattice it seemed advisable to investigate this correlation. This necessitated finding the traces on the surface of the specimen of a plane tangent to the sphere at each of the pairs of spots, thus involving a problem in spherical trigonometry. The equations giving the relation between the angles of latitude, longitude and those made by the traces of the octahedral planes on the specimen with the edges of the specimen, (as shown in Fig. 2) are as follows:

$$c = \arctan (\cot b \ \sin a)$$
$$d = \arctan (\cot b \ \cos a)$$

Knowing these angles for each set of octahedral planes and the load on the specimen, the area of intersection of each plane with the specimen, the angle between the plane and the center line of the specimen and thus the normal and shearing stresses on each of these planes can be calculated by ordinary analytical or graphical methods.

TEST RESULTS

The angles of the slip lines, corresponding to c and d above, were read directly by mounting the specimen on the stage of a low-power microscope in which there were cross-hairs and on which angles of rotation of the stage could be read. By comparing the angles between the slip lines and the edges of the specimen with the angles between the traces of the octahedral planes and the edges of the specimen, the planes on which slip occurred

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[1] H. J. Gough, "Crystalline Structure in Relation to Failure of Metals—Especially by Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 3 (1933), contains an extensive bibliography.

TABLE I.—CREEP TEST RESULTS FOR SINGLE-CRYSTAL SPECIMENS.

The relative positions of octahedral planes are shown in Fig. 3.

Single-crystal Specimen	Load, lb.	Octahedral Plane	Normal Stress, lb. per sq. in.	Resolved Shearing Stress, lb. per sq. in.	Rate of Creep, per cent per hr.	Remarks
A	9.5	(111)	119	147	Less than 0.000018	No creep detected in 984 hr.
		$(\bar{1}11)$	140	152		
		$(\bar{1}1\bar{1})$	101	142		
		$(11\bar{1})$	99	142		
	16.0	(111)	201	248	0.00036	No slip lines were visible after 4661 hr. at this load.
		$(\bar{1}11)$	235	256		
		$(\bar{1}1\bar{1})$	170	240		
		$(11\bar{1})$	165	239		
B	13.0	(111)	132	194	Less than 0.00002	No creep detected in 506 hr.
		$(\bar{1}11)$	338	164		
		$(\bar{1}1\bar{1})$	73	160		
		$(11\bar{1})$	2	32		
	16.5	(111)	165	241	Less than 0.0000063	No creep detected in 1625 hr.
		$(\bar{1}11)$	422	205		
		$(\bar{1}1\bar{1})$	92	200		
		$(11\bar{1})$	3	40		
	22.8	(111)	231	339	0.0025	Slip lines visible on (111) plane. 4661 hr. at this load.
		$(\bar{1}11)$	592	287		
		$(\bar{1}1\bar{1})$	128	280		
		$(11\bar{1})$	3	57		
C	11.2	(111)	202	142	Less than 0.00002	No creep detected in 480 hr.
		$(\bar{1}11)$	108	146		
		$(\bar{1}1\bar{1})$	109	147		
		$(11\bar{1})$	88	137		
	18.8	(111)	337	237	Less than 0.000028	No creep detected in 358 hr.
		$(\bar{1}11)$	180	243		
		$(\bar{1}1\bar{1})$	182	246		
		$(11\bar{1})$	146	229		
	22.2	(111)	400	282	0.000038	Slip lines visible on $(\bar{1}1\bar{1})$ plane. 1850 hr. at this load.
		$(\bar{1}11)$	214	289		
		$(\bar{1}1\bar{1})$	216	292		
		$(11\bar{1})$	173	272		

TABLE II.—CHEMICAL COMPOSITION OF CRYSTALS.

Crystal	Bismuth, per cent	Copper, per cent	Silver, per cent	Antimony, per cent	Tin, per cent
A	0.06	about 0.05	0.006	0.025	0.025
B	0.06	about 0.025	0.006	no lines	no lines
C	0.06	about 0.05	0.006	no lines	no lines

were definitely identified as octahedral planes. Furthermore, they were those octahedral planes on which the resolved shearing stress was a maximum. The term "resolved shearing stress" denotes the shearing stress due to the component of the load along a particular plane.

The new crystals, after having had their orientations determined, were placed in a creep rack under steady tensile stress at room temperature. An extensometer reading to 0.0001 in. was attached over a 1-in. gage length on each test specimen and readings of the strain were taken periodically. Various conditions of loading, stresses and rates of creep are recorded in Table I. From a study of this table it will be noted that, for resolved shearing stresses on the octahedral planes below about 250 lb. per sq. in., creep was not observed and for stresses above this value creep was observed. Table II gives the chemical composition of the three crystals as determined by spectrographic methods.

Strain-time diagrams for one condition of loading on each of the three crystals are shown in Fig. 4. Their general shape and erratic nature are of interest since they contrast so sharply with corresponding diagrams for extruded polycrystalline lead. The strain-time curve for polycrystalline lead is composed of three parts, roughly. First, there is a period following the initial deformation due to the application of the load, in which creep proceeds at a decreasing rate and approaches an approximately constant value. This constant rate prevails during the second stage. In the third and final stage the rate of creep increases rapidly until fracture occurs. The value of the creep rate in the second stage, when it is nearly constant for the greater part of the "life" of the specimen, is generally regarded as of greatest significance and hence is used in Fig. 4 as a basis of comparison with the single-crystal behavior. The marked difference between corresponding diagrams for the single crystals and for the polycrystalline material must be attributed to movement between crystals at the grain boundaries of the polycrystalline metal and to constraining action between the crystals.

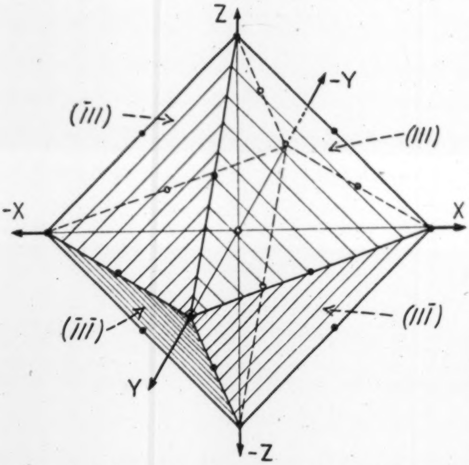

FIG. 3.—A Crystallographic Octahedron Showing Octahedral Planes Listed in Table I.

On removal from the test rack the test specimens were examined under a microscope for slip lines. Crystal *A* which had suffered a 2 per cent elongation showed no slip lines, whereas crystals *B* and *C* each had a well-defined set of slip lines on each of their respective faces. They were much broader on crystal *B* in which the greatest amount of deformation took place.

The absence of visible slip lines on crystal *A* might have been due to the fact that the surface originally was "as cast" and had been subjected to atmospheric corrosion in the laboratory for several months. From Table I it may be seen that the resolved shearing stresses on the four sets of octahedral planes was very nearly the same. This might have caused the slip to be well distributed over the several sets of planes so that an insufficient amount occurred on any one set to leave visible marks on the surface.

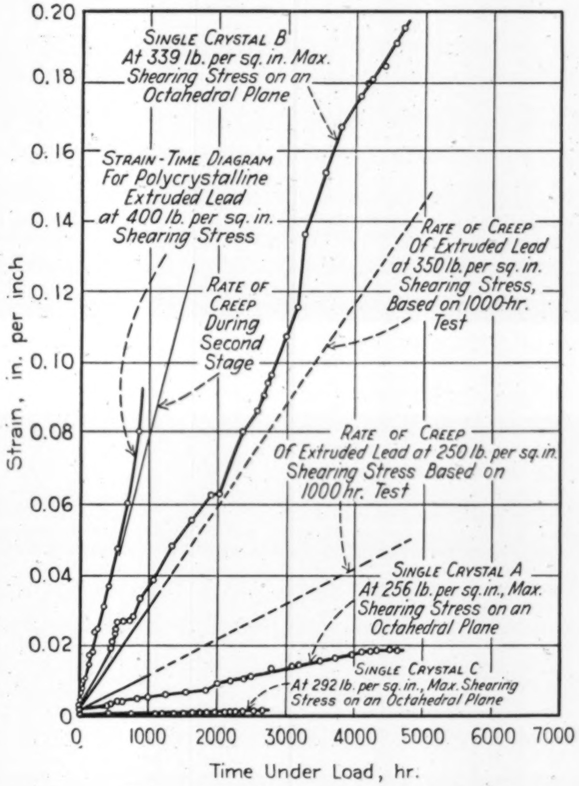

FIG. 4.—Creep of Single-Crystal Specimens of Lead.

SUMMARY AND CONCLUSIONS

An excursion has been made by an engineer into a field of scientific investigation heretofore held quite largely by physicists. The method of

Geo. L. Smith

making the single-crystal specimens and a simple, rapid and reasonably accurate method of determining their crystallographic orientation have been described.

Tests to date show that the strain-time diagrams for single crystals of commercially pure lead are quite different from those of polycrystalline specimens of the same material. In the latter, creep is due partly to deformation within the grains and partly to disturbances at the grain boundaries.

The mechanism of creep in single-crystal specimens of commercially pure lead seems to be by slip on that octahedral plane on which the resolved shearing stress is a maximum.

There are some grounds for believing that there is a limiting resolved shearing stress (approx. 250 lb. per sq. in.) on the octahedral planes below which creep takes place exceedingly slowly, if at all, and above which it may proceed fairly rapidly. This apparent limiting shearing stress is somewhat higher than the lowest shearing stress at which creep has been observed in polycrystalline extruded lead.

DISCUSSION

MR. A. J. PHILLIPS[1] (*presented in written form*).—Mr. Betty has demonstrated very nicely the important role which slip plays in the plastic deformation of lead crystals. I should like to point out that the mere isolation of the plane of slip in a crystal system does not adequately instruct one in the mechanism of slip or permit one to make mathematical deductions or interpretations of the data. It has been found that as a general rule slip takes place not only in definite crystal planes but also in definite crystallographic directions on these planes. Thus, slip may take place on the octahedral plane on which the resolved shearing stress is not a maximum if a slip direction on that plane is more favorable than any slip direction in the plane of maximum shearing stress.

Most metal crystals in the face-centered cubic lattice slip most readily on the (111) plane and in the [110] direction. Now, there are four planes of the (111) form, and in each of these four planes there are three [110] slip directions. Now, as there are only six [110] slip directions in the lattice, it is obvious that the various slip directions are shared by different (111) planes and a study shows that for any given [110] slip direction there are two (111) planes which share this direction. Consequently, it is possible to have slip take place on two (111) planes simultaneously and without interruption, providing, of course, the applied stress is such as would cause this slip. It is thus evident that the mathematical computation of the effect of orientation upon extension of single crystals is a little more complicated than it would seem at first glance. In addition to this simultaneous slip on two planes, there is the further complicating feature of rotation of the crystal due to slip. Naturally as the single crystal is extended the (111) plane on which the slip is taking place tends to revolve into the direction of extension. This, of course, must be accompanied by slip on other planes, at least in certain areas, in order to permit the change in orientation.

It is thus evident that a study to determine the threshold slip for lead crystals must determine not only the slip planes but also the slip directions and the effect of participating slip planes in limiting slip on the favored plane. The absence of slip lines in the surface of a metal crystal does not indicate by any means that slip has not taken place within the crystal for if the slip direction is parallel to the plane of the surface, slip lines will not be seen.

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[1] Superintendent of Research, American Smelting and Refining Co., Maurer, N. J.

I should like to call attention to the fact that the compositions given in Table II of the paper are quite significant. I do not believe that it is permissible to classify creep data on leads containing 0.05 per cent copper with those on leads containing 0.025 per cent copper.

MR. P. G. MCVETTY[1] (*presented in written form*).—The author of this paper has made a valuable contribution not only to our knowledge of the properties of lead and lead alloys but also to a better understanding of creep phenomena which may govern the action of heat-resisting alloys at elevated temperatures.

There has been much speculation as to the nature of creep. A more perfect knowledge of the fundamental conditions involved would do much to aid in investigations of creep properties. The most promising method of attack appears to require a correlation of creep effects with crystalline structure. It would be extremely valuable to know, for example, the relative importance of slip along crystallographic planes within the crystal and flow of the boundary material between crystals. A comparison of creep tests of similar material in single crystal and polycrystalline forms throws much needed light on this important question.

On account of the importance of knowing the relation between the crystallographic orientation and the direction of the applied stress, it is particularly gratifying to see this practical application of the relatively simple Bridgeman method. It is hoped that the future will find further correlation of the closely related fields of the physicist and the engineer.

A comparison of these data with similar results reported by Hanson and Wheeler[2] for aluminum raises several interesting questions. One relates to possible extrapolation of the creep curves. In some cases continued creep leads to a point fracture, while in others failure results from intercrystalline cracking. In such cases, the second stage creep rate may not give a completely satisfactory method of comparison.

Another question arises concerning the uniformity of the polycrystalline specimens. Haigh and Jones[3] found considerable difference in stress-strain diagrams for slow rates of straining depending on method of production and grain size. It would be interesting to know how much variation in creep rate was observed in tests under similar conditions as an aid in interpreting Fig. 4 of the paper.

MR. B. B. BETTY.[4]—I presume Mr. Phillips speaks from experience on the deformation of single crystals in a rapid tension test. When I undertook this investigation very little work had been done toward explaining the mechanism of deformation of crystals under prolonged steady load. It was not known at that time, especially for lead, whether the mechanism of creep deformation properly could be called slip and, if so, on what plane it would take place; nor was it known in what direction slip would occur. Mr. Phillips brought up the subject of the direction of slip on the octahedral planes. This phase of the mechanism of plastic distortion under creep conditions was not mentioned in the body of the paper, since the author did not determine the direction of slip on these planes experimentally, whereas, he did identify the planes on which the slip took place. However, the shearing stresses on the (111) planes in the [110] directions were computed. In each case the (111) planes subjected to the maximum nominal shearing stress contained the [110] direction in which the shearing stress was a maximum, so that I cannot differentiate between the two. I did not have a right to make a definite statement on the matter of direction of slip, since I did not determine it in the laboratory, but in future work I shall be able to do so. It is a little difficult and tedious to determine the direction of slip especially for soft metals like lead.

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[1] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] D. Hanson and M. A. Wheeler, "The Deformation of Metals Under Prolonged Loading," *Journal*, Inst. Metals, Vol. 45, p. 229 (1928).

[3] B. P. Haigh and B. Jones, "Interpretation of the Tensile Test (with Reference to Lead Alloys)," *Journal*, Inst. Metals, Vol. 51, p. 49 (1933).

[4] Research Assistant in Engineering Materials, Engineering Experiment Station, University of Illinois, Urbana, Ill.

Chemical composition has its effect in the creep of polycrystalline metal and in the resistance to the deformation of other single crystals in a rapid tension test, so that we would expect to find a difference in resistance to creep on account of the difference in the chemical composition of these crystals. They are not exactly comparable.

The author is thankful to Mr. McVetty for emphasizing the broader significance of this method of attack in obtaining a better understanding of the creep properties of commercial metals. It is planned, in the near future, to run creep tests of a wide range of grain sizes holding other factors as near constant as is feasible. The author hopes that such tests will provide at least a partial answer to the question of the inherent effect of grain size on rate of creep.

SOME TESTS ON TIN BRONZES AT ELEVATED TEMPERATURES

By J. W. Bolton[1]

Synopsis

The alloy specified in A.S.T.M. Standard Specifications for Steam or Valve Bronze Sand Castings (B 61),[2] an essentially solid-solution structure, is shown to have a "limiting creep strength" of about 8000 lb. per sq. in. at 500 F. and to be free from embrittlement. At 600 F. the alloy has low "creep strength" and is of low ductility after exposure to this temperature for long periods.

The alloy specified in A.S.T.M. Standard Specifications for Sand Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (B 60)[3] (the composition, copper 88 per cent, tin 10 per cent, zinc 2 per cent, falling within this specification is the one specifically referred to) consists of an essentially solid solution matrix, throughout which a high tin component (the alpha-delta eutectoid) is dispersed in small particles. This alloy's load-carrying characteristics at 500 F. are inferior to alloy B-61, and there is evidence of embrittlement under exposure to a temperature of 500 F. Microscopic study, especially of material exposed to a temperature of 600 F., shows clearly that this embrittlement is due to formation and possibly to penetration of the brittle eutectoid structure along grain boundaries.

In this paper there are presented data on "creep" and some other effects of exposure to temperature under sustained loads, on alloys specified in the Society's Standard Specifications for Sand Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (B 60)[3] and in Standard Specifications for Steam or Valve Bronze Sand Castings (B 61).[2] For convenience these two alloys will be referred to as alloys B-60 and B-61.

The alloy B-60 is known to the trade as 88-10-2, 88-8-4, admiralty gun metal, etc. The alloy B-61 is termed steam or valve bronze. It is very widely used in pressure-containing parts subjected to moderately elevated temperatures. Alloy B-60 and related alloys also are used to some extent in industry for such applications.

Recent revisions[4] of specifications B 60 and B 61 call for the following minimum room-temperature physical properties:

	Alloy B-60	Alloy B-61
Tensile strength, lb. per sq. in.	38 000	34 000
Yield point, lb. per sq. in.	16 000	16 000
Elongation in 2 in., per cent	22	22

Note.—These minima are somewhat higher than those specified in the 1933 Book of A.S.T.M. Standards, Part I.

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[1] Chief Chemist and Metallurgist, The Lunkenheimer Co., Cincinnati, Ohio.

[2] 1933 Book of A.S.T.M. Standards, Part I, p. 608.

[3] *Ibid.*, p. 589.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, pp. 1260, 1259 (1934); also 1934 Book of A.S.T.M. Tentative Standards, pp. 1188, 1187.

The object of the work which we conducted was to determine the limiting temperatures within which alloys B-60 and B-61 may be safely applied in pressure-containing parts by ascertaining the ability of these alloys to sustain loads at elevated temperatures and their structural stability under conditions of temperature and loading; that is, "creep" characteristics and freedom from or tendency toward embrittlement, as the case might be. Previous creep tests[1] indicate that alloy B-61 had the better load-carrying ability of the two alloys, and probably could be safely used at temperatures up to 550 F. in conservative design.

Casting Procedure:

For each alloy, a number of bars was cast from a ladle. Green-sand molds were used; the reduced section of the bars was about 0.53 in. in diameter as cast; and the risers were in the gating system. (That is, we did not use the heavy "Webbert type" riser along the top of the bar, although bars were cast in the horizontal position.)

Composition:

The alloys tested had the following composition:

	ALLOY B-60	ALLOY B-61
Copper, per cent	87.60	87.14
Tin, per cent	10.18	6.22
Zinc, per cent	2.02	4.61
Lead, per cent	none	1.83

Impurities are held to low limits.

Room Temperature Tests:

The following are results of tests conducted at room temperatures:

	ALLOY B-60	ALLOY B-61
Diameter tested, in	0.505	0.505
Tensile strength, lb. per sq. in	49 100	37 800
Yield point, lb. per sq. in	20 400	17 200
Elongation in 2 in., per cent	35.4	34.3

Tests on Creep Bars:

Bars 0.357 in. in diameter were used. These were cut from bars 0.53 in. in diameter as cast. The temperature uniformity calibration, loading accuracy, etc., complied with the A.S.T.M. Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 - 34 T).[2] The Lunkenheimer Co. equipment and calibration methods have been described previously.[3]

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[1] Symposium on Effect of Temperature on the Properties of Metals, published jointly by the American Society for Testing Materials and The American Society of Mechanical Engineers, pp. 361–363 (1931). Available as separate publication.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1223 (1934); also 1934 Book of A.S.T.M. Tentative Standards, p. 1147.

[3] H. Montgomery and J. W. Bolton, "Furnaces for Elevated Temperature Tests," *Metals and Alloys*, June, 1934, pp. 127–128.

After testing in creep, the bars were removed from the furnaces, and tested in tension at room temperature.

TEST RESULTS

Alloy B-60 (88-10-2):

Careful inspection of Fig. 1 shows continued deformation at all combinations of temperatures and loads chosen. Taking the last 1200 hr. of test on bars *B*, *D*, and *E*, the following rates are shown:

	Temperature, deg. Fahr.	Load, lb. per sq. in.	Rate, per cent in 10,000 hr.
Bar *B*	500	6000	0.29
Bar *D*	550	4000	0.20
Bar *E*	600	3000	0.45

After removing from the creep test the bars were tested in tension at room temperature with the following results:

	Temperature, deg. Fahr.	Creep Load, lb. per sq. in.	Duration, hr.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent
Bar *A*	500	6000	1030	43 200	26.7
Bar *B*	500	6000	1700	39 400	19.8
Bar *C*	500	8000	700	38 900	17.6
Bar *D*	550	4000	1700	38 000	17.2
Bar *E*	600	3000	1700	33 200	10.7
Original	room	none	....	49 100	35.4

In all cases there occurred a marked drop in strength and ductility despite the fact that the permanent or "creep" deformations in no case in themselves account for much of the loss of elongation. Bars *A* and *B* are interesting in that increased duration of exposure to temperature in itself lowers strength and ductility, the temperature and load being constant. The table suggests that the high-tin alloy containing alpha-delta eutectoid becomes embrittled on long exposure to temperatures 500 F. or above, particularly if loaded high enough for some creep to occur. In case of bar *E* (loaded at 600 F., then broken at room temperature) a very marked coarsening of fracture was observed.

Alloy B-61 (Valve or Steam Bronze):

It will be noted that in bars *A* and *B*, Fig. 2, the loads were stepped up from 6000 lb. per sq. in. and 8000 lb. per sq. in. to 7000 lb. per sq. in. and 9000 lb. per sq. in., respectively, after 530 hr. of testing. No creep is shown in bar *A*. In bar *B* there is slight creep and on bar *C* loaded to 10,000 lb. per sq. in. there is quite rapid creep.

The results are shown below, creep rate being ascertained during the period beyond 500 hr. at the loads indicated:

	Temperature, deg. Fahr.	Load, lb. per sq. in.	Rate, per cent in 10,000 hr.
Bar *A*	500	7 000	nil
Bar *B*	500	9 000	0.18
Bar *C*	500	10 000	1.00
Bar *D*	550	5 000	0.28
Bar *E*	600	4 000	1.77

After creep testing, the bars were removed and pulled in tension at room temperature with the following results:

	Temperature, deg. Fahr.	Creep Load, lb. per sq. in.	Duration, hr., total	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent
Bar *A*	500	6 000 to 7 000	2100	37 750	38.7
Bar *B*	500	8 000 to 9 000	2100	38 000	36.7
Bar *C*	500	10 000	1150	31 800	25.8
Bar *D*	550	5 000	1180	33 200	21.8
Bar *E*	600	4 000	1180	17 000	8.1
Original	room	none	....	37 800	34.3

The results shown are of considerable interest. After test at 500 F. at loads within or slightly above a limiting creep rate of 0.1 per cent in 10,000 hr. (up to 9000 lb. per sq. in.) *there is no loss in strength or ductility* shown by subsequently testing at room temperature. (This loading, or stress, is far above that contemplated in valve design.) At loads producing rather rapid creep (bar *C*) appreciable loss in strength and ductility results. At 550 F., at a load (5000 lb. per sq. in.) producing a creep rate of 0.28 per cent per 10,000 hr., some loss in strength and ductility results, but nothing like that observed at a lower loading (4000 lb. per sq. in.) in the case of alloy B-60 (88-10-2). It is readily conceivable that at lower loads alloy B-61 at 550 F. may be unimpaired in strength and ductility. At 600 F. there is a very great loss of strength and ductility, probably due as much to exposure to temperature as to the choice of a load producing a relatively rapid creep rate. It may be mentioned, however, that the coarse grain formation produced in alloy B-60 (88-10-2) at 600 F. was not noted in Alloy B-61.

Comparisons of Alloys B-60 and B-61:

It is obvious that there is a fundamental difference between alloys B-60 and B-61 in their behavior under temperature. At 500 F., and probably even at 550 F., there should be no hesitancy in specifying alloy B-61 for stresses within those required to produce 0.1 per cent secondary creep rate per 10,000 hr. It appears that the use of alloy B-60 at 500 F. should be confined to applications where design is conservative and stresses are low. In no case does it seem conservative engineering practice to employ alloy B-60 at temperatures above 500 F.

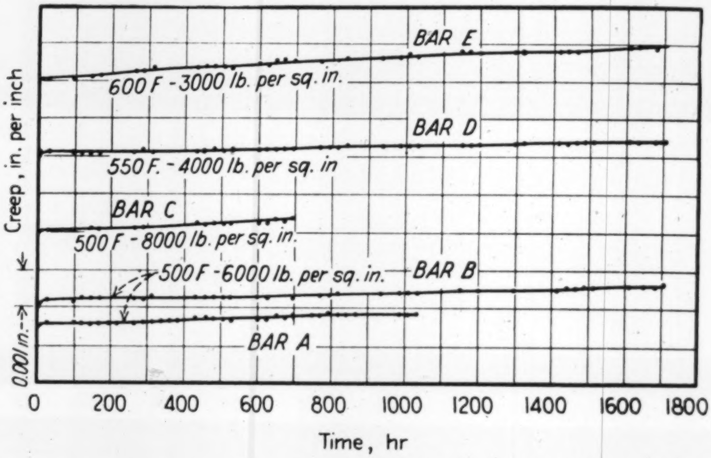


FIG. 1.—Creep Test on Alloy B-60 (88-10-2).

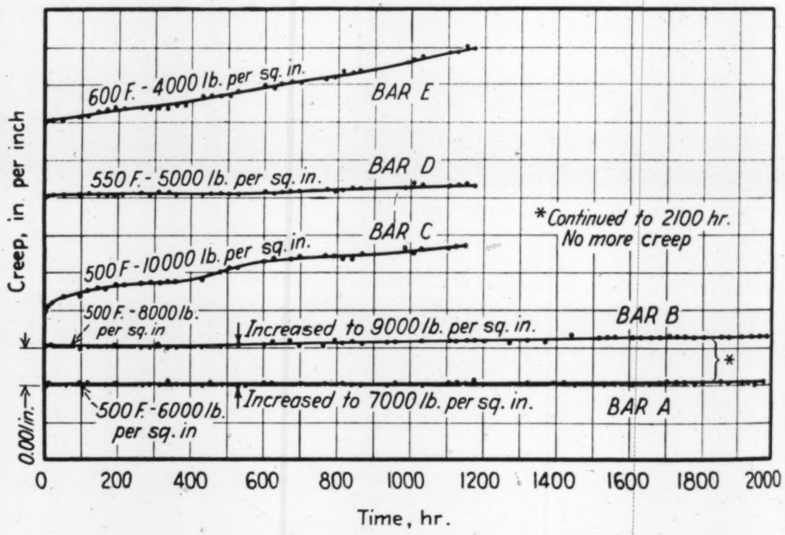


FIG. 2.—Creep Test on Alloy B-61 (Valve Bronze).

The question may well be asked why there should be this great difference in behavior at temperatures around 500 to 550 F. The tin in alloy B-61 is present in solid solution with the zinc and copper. In alloy B-60, in commercial casting practice, some of the tin is present in the alpha-delta eutectoid form, as isolated tin-rich particles. The practical foundryman, familiar with the phenomenon of "tin sweat," will have little difficulty in visualizing the possibilities. Whatever speculations may be indulged in, it is evident that absence of a tin-rich component (as found in higher tin alloys) is advantageous.

Microscopic study was made on the samples removed from test at 600 F. and on other samples. The sections chosen for micro-examination were in the heads of the test bars, where distortion is at a minimum. The general microstructures of alloys B-60 and B-61 as cast are well known. Alloy B-60 has a cored structure, essentially a solid-solution matrix, with small particles of the tin-rich alpha-delta eutectoid—alloy B-61 has a cored structure, essentially a solid solution, with small lead droplets, but no apparent alpha-delta eutectoid.

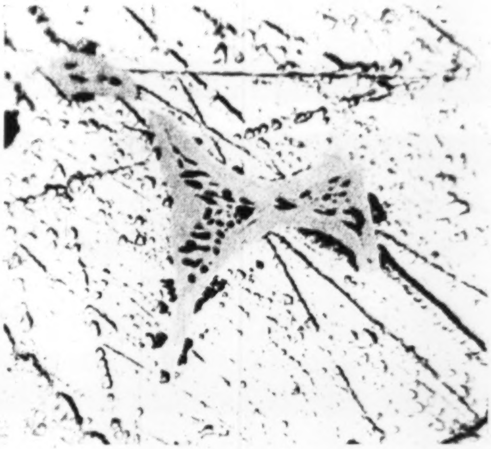
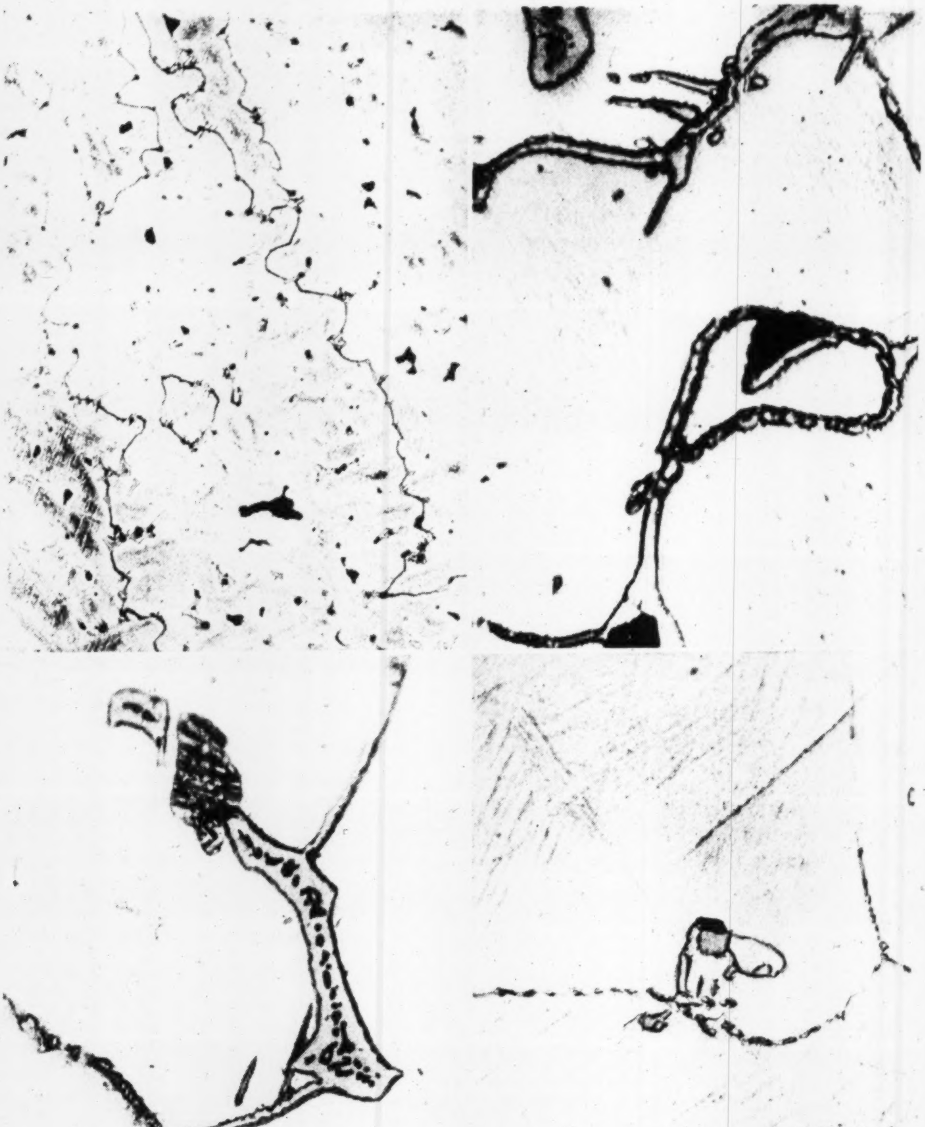


FIG. 3.—Delta Eutectoid of Alloy B-60 (× 1000).
Etched with acidulated ferric chloride.

In alloy B-60 the alpha-delta eutectoid is much harder than the matrix. Figure 3 shows a particle of this eutectoid with the matrix purposely left scratched up to show the hardness difference clearly.

Figure 4 (*a*) shows the general structure of the alloy B-60 bar (head) after being removed from test at 600 F. A secondary grain formation surrounded by a network is apparent. Close inspection shows that the network extends between various eutectoid particles. Higher magnifications, Fig. 4 (*b*) and (*c*), show that the networks are formed by formation and perhaps to some degree penetration of the alpha-delta eutectoid component along grain boundaries. The blue-gray color of the network as contrasted with the yellow color of the matrix is very clear on visual examination.

The sample of alloy B-61 which had been tested at 600 F. is shown. Whereas alloy B-60 (88-10-2) was completely penetrated by networks,



(*a*) Alloy B-60 (× 100). (*b*) Alloy B-60 (× 1000).

(*c*) Alloy B-60 (× 1000). (*d*) Alloy B-61 (× 1000).

FIG. 4.—Photomicrographs of Alloys B-60 and B-61.
Etched with ammoniacal hydrogen peroxide.

alloy B-61 had but small isolated areas so affected. The worst spot is shown in Fig. 4 (*d*). It will be noted that the small particles along the boundaries are not continuous. Only small areas were so affected.

The samples of alloys B-60 and B-61 tested at 500 F. and 550 F. also were examined. Alloy B-60 (88-10-2) showed marked tendency toward network formation in the sample tested at 500 F. and very distinct network formation at 550 F. Alloy B-61 showed no formation at 500 F. nor were such formations perceptible at 530 F.

From the above studies it is evident that the alpha-delta eutectoid when present acts as an embrittling agent at elevated temperatures and bronzes containing this eutectoid should be viewed with suspicion for applications at 500 F. and above. On the other hand bronzes free from this eutectoid and which do not precipitate this eutectoid when held at elevated temperatures do not show embrittlement and should be regarded as a separate species. At 500 F., or probably somewhat above, alloys with about 6.2 per cent tin and 4.6 per cent zinc do not form this eutectoid. At 600 F. such bronzes show precipitation of a small amount of the eutectoid. With tin content constant, the formation of the eutectoid may be prevented by some restriction of the zinc content (say to 4 per cent) or by the addition of small percentages of nickel (say about 0.7 to 1.0 per cent). In commercial experience, at room temperatures, the eutectoid is not found up to 6.5 per cent tin (or possibly slightly above) when the zinc content is within the limits of Specifications B 61.

Although the samples studied had been subjected to effects of stress as well as to effects of temperature, it is the author's opinion that the marked changes noted in microstructure after creep testing are due primarily to the exposure to temperature—the effects of stress being secondary. At high stresses both the effects of such stresses and of temperature appear to affect ductility.

CONCLUSION

It is the author's opinion that bronze of the alloy B-61 type is very well suited for use at 500 F. and possibly up to 550 F. It is unsuited to applications where the temperature of the metal structure reaches 600 F. The design stress at 500 F. should be limited to 5000 lb. per sq. in. It has been shown that at 500 F. alloy B-61 has a "limiting creep strength" (based on that stress which produces a rate of flow less than 0.1 per cent in 10,000 hr. after the first 500 hr. of creep tests, running up to 2100 hr. duration) of about 8000 lb. per sq. in., or about 1.6 times the recommended maximum design stress. These results suggest that it would be in order to indicate this temperature limit as practical and conservative, in an early paragraph in Specifications B 61.

In case of bronzes containing the alpha-delta eutectoid including the compositions contemplated by Specification B 60, and especially the high-tin alloy 88-10-2, the tests run indicate that at 500 F. the load-carrying ability is lower than that of alloy B-61, and there is distinct evidence of embrittlement. Microscopic study suggests that this embrittlement is caused by the formation and to some degree possibly to penetration of the eutectoid along grain boundaries. This condition becomes very apparent at 600 F. It is the author's opinion that from a code and specification viewpoint the use of such high tin alloys probably should be confined to 450 F. and permissible stress of 5000 lb. per sq. in. at that temperature. He is cognizant of the fact that such alloys have been used with apparent success. It is possible that despite the apparent operating temperatures, the actual temperatures at the metal structure may be lower than the operating temperature.

Acknowledgment:

The author is indebted to The Lunkenheimer Co. for permission to carry out this work and to publish the results; also to Mr. H. Montgomery for the creep tests. Due to a very heavy general development program, it has not been possible to include other alloys or to go as far as we had hoped. However, further data are being accumulated.

DISCUSSION

Mr. H. W. Maack.[1]—Information such as that presented by Mr. Bolton is urgently needed in connection with the design and service rating of products made of bronzes of the type discussed. A search of the literature will reveal a surprising scarcity of information on this subject.

The laboratory with which the speaker is connected has done some work along parallel lines. The accompanying Fig. 1 shows the time-elongation curves for three A.S.T.M. bronzes. The top one is for alloy B-62, commonly known as 85-5-5-5, ounce metal or composition brass. The middle curve is for alloy B-61 or valve bronze, and the bottom one is for alloy B-60, 88-10-2 bronze.

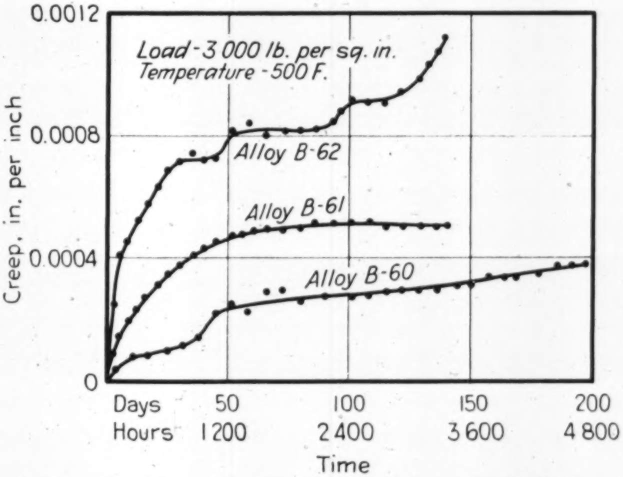


Fig. 1.—Time-Elongation Curves for Three A.S.T.M. Bronzes.

Examination of the curves shows that alloy B-62 creeps at a considerably more rapid rate than either alloy B-61 or alloy B-60 at this temperature of 500 F. At 3000 lb. per sq. in. stress, alloy B-61 shows practically no creep in the period from 1500 to 3336 hr., whereas alloy B-60 creeps at a rate of about 0.039 per cent per 10,000 hr. for the period from about 1200 hr. to 4728 hr. Alloy B-62 shows a nil creep rate for the period from 1200 to 2000 hr., whereupon the creep increased to 0.44 per cent per 10,000 hr. after 3000 hr. of testing. These data covering alloys B-60 and B-61 confirm Mr. Bolton's findings that alloy B-61 is much more resistant to creep than alloy B-60.

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[1] Chief Chemist and Metallurgist, Crane Co., Chicago, Ill.

In connection with determinations of the creep strength of metals, it is interesting to consider also temperature effects in service applications of the metals or alloys. Differences of considerable magnitude frequently exist between line temperature and actual metal temperature. These often constitute an added factor of strength.

The accompanying Fig. 2 shows a $1\frac{1}{2}$-in. bronze globe valve with thermocouples attached for measurement of temperature differences. Couples for the determination of metal temperature have their junction peened into saw cuts in the bronze. The junction of the couple for measuring steam temperature is at the center of the port at the inlet end of the valve. Installed uninsulated in a superheated steam line, with the valve stem or spindle vertical, temperature readings were as follows: steam 500 F.; bridge 483 F.; side of body 480 F.; bottom of body 470 F. The temperature gradient between inside and outside surfaces of the body wall of an uninsulated valve is probably of considerable consequence also.

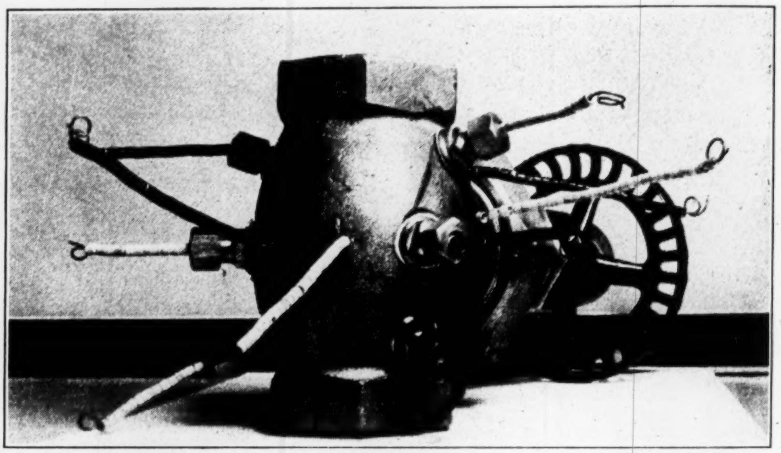

FIG. 2.—Bronze Globe Valve with Thermocouples Attached for Measurement of Temperature Differences.

Taking into consideration the 30-deg. difference between steam at 500 F. and metal temperature of 470 F. at the bottom of the valve in its effect on the creep strength of bronze, one finds a difference of about 3000 lb. per sq. in. or roughly one third the creep strength of the bronze at 500 F. Obviously, favorable temperature differences and metal temperature gradients add much to the factor of safety of metals in service.

MR. T. E. KIHLGREN[1] (*presented in written form*).—The author has obtained some interesting data on two types of bronze foundry mixtures which are widely used. We should like, however, to call attention to a few points which may be open to criticism.

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[1] Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

In discussing the structural changes occurring in the tensile bars after test, a clearer discrimination between the alpha-delta eutectoid and the phases constituting it would leave less chance for confusion. In his discussion of the 88-10-2 alloy (alloy B-60) after exposure at 600 F. (photomicrographs Figs. 4 (*a*), (*b*) and (*c*)), the author refers to a network, extending between patches of eutectoid, which he describes as due to "formation and perhaps to some degree penetration of the alpha-delta eutectoid component along grain boundaries." If the author implies that the network consists of the delta phase rejected from the eutectoid patches present in the "as cast" condition, we should not find ourselves in agreement. Actually, it would seem that the network consists of the delta phase precipitated (as delta) from a supersaturated alpha solid solution after soaking for a long period at 600 F. Similarly, the lower tin content alloy B-61 shows little of this type of embrittlement, probably because the alpha solid solution is less supersaturated at that temperature and the amount of delta rejected from the alpha solid solution is therefore relatively small. If this view were accepted, we should then be forced to disagree with the statement on page 211, that is, "the alpha-delta eutectoid when present acts as an embrittling agent, etc."

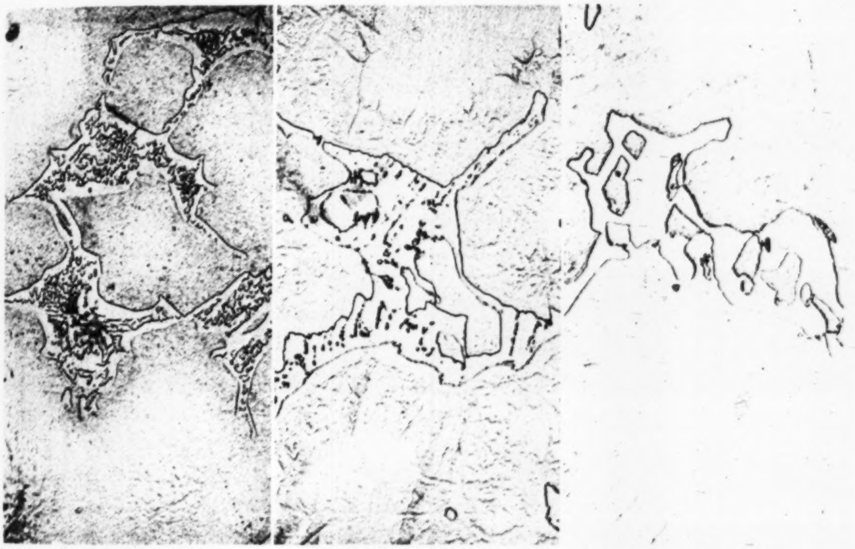
(*a*) 0.02 per cent Nickel. (*b*) 1.47 per cent Nickel. (*c*) 2.94 per cent Nickel.

FIG. 3.—Effect of Nickel on the Structure of Sand-Cast 89-11 Gear Bronze (× 500). As Cast.

The fact that the delta phase is precipitated along grain boundaries indicates that the castings are higher in tin in these regions, and further suggests that castings of the 88-10-2 type when used at elevated temperatures might be expected to perform better if they are subjected to a homogenizing anneal at some temperature as 1400 F. before placing in service.

A strict adherence to proper terminology with respect to the structural constituents present in these alloys is particularly important when the role of such elements as nickel in suppressing the formation of the eutectoid is discussed, if the data are to be correctly interpreted. The effect of nickel on the structure of sand cast 89-11 gear bronze is shown in the accompanying Fig. 3 (500 diameters) in which 0, 1½ and 3 per cent of nickel has been substituted for copper. It may be seen that nickel suppresses the formation of the (alpha + delta) eutectoid and at 3 per cent nickel, the eutectoidal structure has entirely disappeared. The effect of nickel on the constitution of simple tin bronzes has been discussed at some length by J. T. Eash and C. Upthegrove[1] who have indicated the alpha-delta eutectoid is replaced by a new phase—"theta." It would be interesting if the author were to present similar creep data and microstructural evidence dealing with the effect of such elements as nickel and zinc on the behavior of these alloys.

The abrupt drop in tensile properties at room temperature observed in the steam bronze after exposure at 600 F. may be due to the presence of molten lead at the elevated temperature. Veszelka[2] in his studies of the copper-tin-lead alloys points out that the lead phase is molten at 606 F., a fact of some significance in view of the sharp drop in strength and ductility at room temperature as the creep temperature was increased from 550 to 600 F. The effect of lead might be readily checked by similar tests in a lead-free bronze of comparable tin content.

MR. E. R. DARBY.[3]—I cannot help but feel that in connection with Mr. Kihlgren's remarks with respect to the 88-10-2 alloy, it would be very interesting to know what the creep properties at elevated temperatures would be if the alloy had been subjected to a short-time high-temperature anneal before the creep tests were started. The alloy, of course, is not in equilibrium as cast and even at temperatures as low as Mr. Bolton made his tests, there is some tendency to assume equilibrium conditions. If we were to assist that, in the beginning, before the test, it might be possible that a new picture could be had.

Annealing this alloy at temperatures between 1200 and 1400 F. causes considerable change in physical properties. After heating for an hour strength and elongation are greatly improved though at the expense, to some extent, of hardness. Extended heating, as long as six hours, gives additional improvement in strength and ductility without much additional change in hardness.

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[1] J. T. Eash and C. Upthegrove, "Copper-Rich Alloys of the Copper-Nickel-Tin System," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 104, p. 221 (1933).

[2] J. Veszelka, "Investigation of Equilibrium Relationships of Further Alloyed Bronzes," *Communications*, Royal Hungarian Mining and Forestry College, Mining and Metallurgical Dept., Vol. 4, p. 186 (1932).

[3] Metallurgist, Federal-Mogul Corp., Detroit, Mich.

With these facts in mind I should like to ask Mr. Bolton if, under such conditions of preliminary treatment, he would expect any different results from creep tests at elevated temperatures.

MR. J. W. BOLTON.[1]—The essential conclusions of Mr. Maack are the same as ours, although he approaches some parts of the problem in a little different manner than we did.

Mr. Darby points out, and rightly so, that the alloys under consideration are not at all in equilibrium. It is problematical, indeed, what a treatment to produce a homogeneous structure would reveal, and Mr. Darby and Mr. Kihlgren both are to be thanked for that suggestion. Mr. Kihlgren's discussion really boils down to three elements, one relative to the phraseology, where he objects to the phrase "this embrittlement is due to the penetration or possibly the formation of the eutectoid along grain boundaries." I really believe from a personal examination of these materials, that not only has the formation of the eutectoid a precipitation hardening effect (using that term in a rather loose sense), but there actually is a partial penetration. If you study some of these photomicrographs, for example, on page 210, and if you could have had the opportunity of looking at a sample, there is in some cases noted a distinct semivoid along some of the sides of the alpha-delta eutectoid particles. It seems that in some cases that component did push out into the grain boundary, although without a doubt some of the effect is due to precipitation hardening action.

Mr. Kihlgren mentions the possibility that the drop in the B-61 alloy's properties may be due to the presence of lead, since other investigators have found that at 600 F. lead in alloys is molten. We have had experience with other types of alloys—rather high copper alloys—with rather substantial percentages of lead. Such alloys were drawn for long periods at as high as 1600 F., and that treatment did not result in lead penetration. I could understand that possible action if the final tension tests were conducted at 600 F. but in view of what we actually have proved in certain other alloys, I am rather doubtful that Mr. Kihlgren's explanation is correct. In the illustration, page 210, Fig. 4 (*b*), is shown a case where there is actual precipitation of the alpha-delta eutectoid, or something closely approaching it in the lower tin alloy B-61. That alloy is not uniform in composition, microscopically speaking, and quite conceivably the local concentration and exposure to temperature causes precipitation in local areas.

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CREEP CHARACTERISTICS OF ALUMINUM ALLOYS

BY R. R. KENNEDY[1]

SYNOPSIS

Aluminum alloys are widely used in a variety of applications in which they are exposed to elevated temperatures. The short-time tensile properties at these temperatures have been studied by a number of investigators, but little attention has been paid to the long-time tensile properties or creep characteristics.

Nine aluminum alloys that have been used or proposed for use at elevated temperatures were selected for investigation. The creep characteristics of these alloys were determined at 400 and 600 F. (205 and 315 C.).

The creep rates of the alloys varied over a wide range. Sand-cast aluminum-copper-nickel-magnesium alloy had the best creep characteristics of the alloys tested. Some of the newer wrought and cast alloys were markedly inferior to this alloy in that respect, although their mechanical properties at room temperature were superior in some cases and they have better casting and forging properties.

The behavior of metals at elevated temperatures has become a subject for study by an increasing number of metallurgists and engineers during the last decade. Metals are today employed in many structures which operate at temperatures much higher than atmospheric, so an accurate knowledge of the mechanical properties of these metals at operating temperatures is of prime importance. The results of short-time tension tests have been of value, but metals may fail at elevated temperatures when stressed to values which might be considered safe when only the short-time tensile properties are considered. Familiarity with the long-time tensile properties or creep characteristics of metals is of value if structures are to operate at elevated temperatures without danger of failure and at the same time without the use of excessive amounts of material.

As the ferrous metals are most widely used in high-temperature installations, the creep characteristics of numerous ferrous alloys have been determined. The aluminum alloys have not been so thoroughly investigated, though Tapsell(**1**)[2] reports the results of a few tests. Due to their relatively low melting points, aluminum alloys are not suitable for use under stress at temperatures much higher than 600 F. (315 C.). At lower temperatures, aluminum alloys find wide application in installations such as internal-combustion engines where light weight and high heat conductivity are of advantage.

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[1] Associate Metallurgist, U. S. Army Air Corps, Wright Field, Dayton, Ohio.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 230.

The effect of elevated temperatures on the properties of aluminum alloys has received considerable attention. Boegehold and Johnson(2) have discussed the uses of aluminum alloys in internal-combustion engines. A number of investigators(3,4,5,6,7) have reported the results of short-time tests at elevated temperatures. The author(8) has studied the effect of elevated temperatures on the strength of aluminum alloys.

Templin, Paul, Braglio and Marsh(4,6) have shown that the strength of aluminum alloys at elevated temperatures as determined by short-time tests is lowered as the time of heating before testing is increased. This change in tensile strength takes place rapidly during the early part of the heating cycle and then much more slowly, but a period of months is sometimes required before complete stability is attained. It was considered desirable to heat specimens for creep tests through the period of rapid change of mechanical properties before the load was applied, but the period of heating was not long enough to produce complete stability. Therefore, when the results of creep tests are compared with those of short-time tests, it should be remembered that the short-time properties are not constant, but would be lower at the end of a long-time test than at the beginning. In determining the creep characteristics of aluminum alloys for use in structures which would be expected to operate for a period of years at elevated temperatures, the use of specimens which had been completely stabilized at testing temperatures might be desirable. This investigation was conducted, however, with reference to aluminum alloys for use in aircraft engines. A military engine has a life in the neighborhood of 1000 flying hours. Commercial engines may be operated for somewhat longer periods, but in no case is the life comparable with that of such structures as steam boilers which must operate for years without failure. The long time-test should indicate the creep characteristics of alloys during the life of an airplane engine.

The maximum temperatures to which aluminum alloy aircraft engine parts are subjected has been reported by Johnson(2) as ranging from 450 F. (230 C.) for liquid-cooled cylinder heads to 650 F. (345 C.) for pistons. In this investigation the creep characteristics of the alloys were determined at 400 F. (205 C.) and at 600 F. (315 C.), the lower temperature representing normal operating conditions and the higher temperature representing extreme conditions. Specimens for long-time tests at 400 F. (205 C.) were stabilized for 68 hr. while those for tests at 600 F. (315 C.) were stabilized for 20 hr. at testing temperature before the tests were started. Short-time tests at the same temperatures were run on certain of the alloys. The specimens for these tests were given the same stabilizing treatments as the creep test specimens, so the short-time properties reported represent the strength of the alloys at the time when creep tests were started.

DESCRIPTION OF APPARATUS

The apparatus used, shown in Fig. 1, was quite similar to that employed by Gillett and Cross(9). The specimens were heated in electric furnaces, *A*, and loads were applied through levers, *B*, by weights, *C*. The specimens used, shown in Fig. 2, had a gage length 0.505 in. in diameter by 2 in. in length. Holes were drilled in the ends of the specimens extending to the gage length for the insertion of thermocouples. The temperature was controlled by means of Leeds & Northrup Micromax Recorder-Controllers one of which is shown at *D* in Fig. 1. In these instruments the dry cells are automatically balanced against the standard cell at 45-min. intervals, so that errors introduced by changes in electromotive force of the dry cells due to variation in room temperature are negligible.

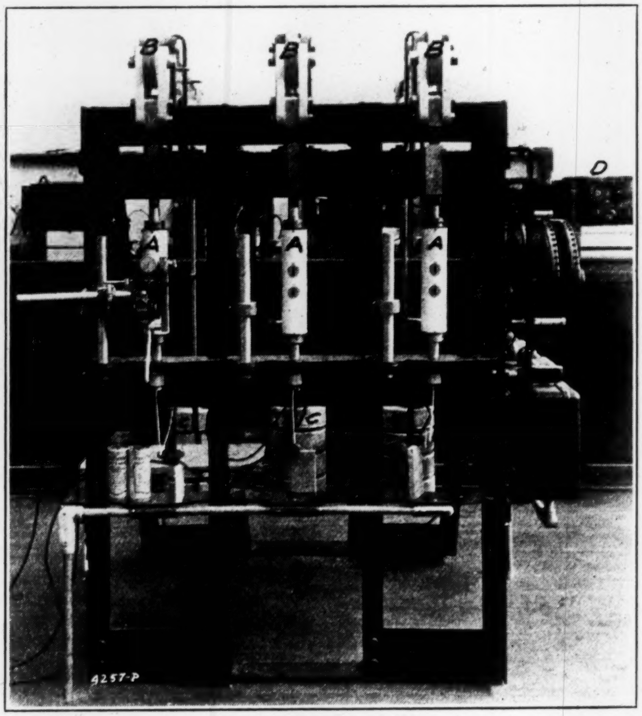

FIG. 1.—Creep Test Apparatus.

The furnaces were of the gap-wound type and contained silver sleeves between the heating elements and the specimens to give more uniform temperature distribution. Thermocouples were inserted in the ends of the specimens, the tips of the couples being located at the ends of the gage length. The top couple was used for reading the temperature of the furnace which was controlled through the lower couple. The hole containing the control couple also contained a loop of chromel wire. When the full current was flowing through the furnace windings, a small amount of current flowed through the chromel loop to supply heat to the thermocouple and bring it up to the control temperature at the same time as the rest of the furnace, thus preventing overshooting. When the control temperature was reached, relays cut off the current to the chromel loop and a portion of the current to the furnace windings though some current continued to flow through the furnace windings to prevent a rapid drop in temperature. The current input to the furnaces could be varied by means of rheostats as the amount of current required varied with the temperature of the furnace.

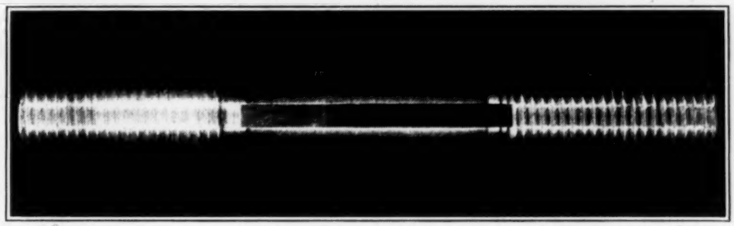

FIG. 2.—Creep Test Specimen.

Temperature exploration of the furnaces under testing conditions indicates that the maximum temperature variation along the gage length of the specimens was ±2 deg. Fahr. (1 deg. Cent.).

The optical device used for determining the extension of the specimens is shown at *E* in Fig. 1. It consisted of a microscope equipped with a 48-mm. objective and a filar eyepiece. The optical device was calibrated against a stage micrometer which had been checked by measurements in a Zeiss Universal Measuring Microscope. The smallest division which could be read with the optical device was 0.000014 in. The sensitivity (the smallest reproducible quantity in terms of units of the gage length) was approximately 40 parts per million. A small illuminator equipped with an electric bulb was clamped to the end of the objective. As shown in Fig. 2, a platinum strip was wired securely to the shoulder at one end of the gage length of the specimen, the free end of the strip extending to the shoulder at the opposite end of the gage length. Small square impressions were made by means of a Vickers hardness tester at the free end of the strip and on the shoulder of the specimen near the free end of the strip. The specimens were mounted in the furnaces so that the free end of the platinum strip and the shoulder of the specimen were opposite the lower

TABLE I.—CHEMICAL COMPOSITION AND HEAT TREATMENT OF ALUMINUM ALLOYS USED.

Alloy	Chemical Composition, per cent						Solution Treatment[a]		Aging Treatment	
	Copper	Iron	Silicon	Magnesium	Nickel	Manganese	Temperature	Time, hr.	Temperature	Time, hr.
No. 1[b]	3.90	0.38	0.21	1.60	2.22	....	950 F. (510 C.)	5	375 F. (190 C.)	5
No. 1-A[c]	3.91	0.35	0.13	1.68	2.22	....	950 F. (510 C.)	5	375 F. (190 C.)	5
No. 2[b]	9.64	1.08	0.22	0.23	....	....	925 F. (495 C.)	5	375 F. (190 C.)	5
No. 3[b]	1.18	0.26	4.61	0.58	....	....	980 F. (525 C.)	16	440 F. (225 C.)	8
No. 4[b]	4.29	0.50	0.66	....	....	....	960 F. (515 C.)	24	300 F. (150 C.)	2
No. 5[c, d]	0.90	0.78	12.64	0.96	0.91	0.02	....	....	....	....
No. 6[c, d]	2.96	0.53	13.18	1.04	3.08	....	....	....	....	....
No. 7[c, d]	4.06	0.62	0.21	0.47	2.12	0.01	....	....	....	....
No. 8[b]	0.37	0.28	0.19	5.96	1.49	1.00	none	....	....	....

[a] Specimens quenched in cold water at conclusion of solution treatment.
[b] Cast alloys.
[c] Wrought alloys.
[d] Alloys supplied in heat-treated condition. Method of heat treatment was not reported.

TABLE II.—RESULTS OF SHORT-TIME TESTS OF ALUMINUM ALLOYS.

Alloy	Temperature of Test[a]	Proportional Limit, lb. per sq. in.	Yield Strength, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Brinell Hardness
No. 1	Room (1)	26 000	......	39 100	1.0	108
	400 F. (205 C.) (1-2)	13 500	......	31 520	1.2	....
	600 F. (315 C.) (1-3)	4 880	......	13 360	6.0	....
No. 1-A	Room (1)	......	52 800	68 530	13.0	
	400 F. (205 C.) (4-5)	22 000	37 250	43 300	10.8	138
	600 F. (315 C.) (4-6)	......	......	6 400	59.0	....
No. 2	Room (1)	34 000	......	42 340	0.5	130
	400 F. (205 C.) (1-2)	12 000	......	28 630	1.5	....
	600 F. (315 C.) (1-3)	2 750	......	10 950	12.5	....
No. 3	Room (1)	9 000	33 470	36 280	1.5	86
	400 F. (205 C.) (1-2)	10 000	......	26 300	2.0	....
	600 F. (315 C.) (1-3)	3 750	......	7 440	13.0	....
No. 4	Room (1)	11 160	23 850	34 030	3.5	87
	400 F. (205 C.) (1-2)	7 500	......	22 830	6.0	....
	600 F. (315 C.) (1-3)	3 750	......	7 990	25.5	....
No. 5	Room (4)	28 000	44 450	52 800	6.0	126
	400 F. (205 C.) (4-5)	16 000	29 250	35 160	4.0	....
	600 F. (315 C.) (4-7)	3 000	6 100	8 710	35.0	....
No. 6	Room (1)	30 000	49 330	59 320	4.0	139
	400 F. (205 C.) (4-8)	......	......	21 700	12.0	....
	600 F. (315 C.) (4-6)	......	......	7 900	45.0	....
No. 7	Room (4)	......	......	51 200	18.7	87
	400 F. (205 C.) (4-9)	......	......	17 500	37.3	....
	600 F. (315 C.) (4-10)	......	......	5 500	72.7	....
No. 8	Room (4)	9 000	18 250	27 270	2.5	74
	400 F. (205 C.) (4-5)	10 000	17 250	23 980	3.0	....

[a] The numbers in parentheses refer to the following notes:

1. Specimens from same lot of metal as that used for creep tests. Tested at Wright Field. The yield strength was taken from stress-strain curves at a deflection of 0.002 in. per inch from the modulus line.
2. Tested after heating to 400 F. (205 C.) for 68 hr.
3. Tested after heating to 600 F. (315 C.) for 20 hr.
4. Test results reported in private communication from Aluminum Company of America on same type of alloy as that used in creep tests.
5. Tested after heating to 400 F. (205 C.) for 1 day.
6. Tested after heating to 600 F. (315 C.) for 75 days.
7. Tested after heating to 600 F. (315 C.) for 1 day.
8. Tested after heating to 400 F. (205 C.) for 75 days.
9. Tested after heating to 400 F. (205 C.) for 30 days.
10. Tested after heating to 600 F. (315 C.) for 30 days.

TABLE III.—CREEP RATES OF ALUMINUM ALLOYS.

Alloy	Temperature	Load, lb. per sq. in.	Total Extension During Testing Period, in. per inch	Duration of Test, hr.	Secondary Creep Rate, per cent per 1000 hr.	Remarks
No. 1	400 F. (205 C.)	2 500	0.000083	313	0.00	
	400 F. (205 C.)	5 000	0.000124	312	0.01	
	400 F. (205 C.)	10 000	0.000206	503	0.02	
	400 F. (205 C.)	12 500	0.000388	532	0.05	
	400 F. (205 C.)	15 000	0.000512	502	0.06	
	400 F. (205 C.)	17 500	0.000735	460	0.12	
	400 F. (205 C.)	20 000	0.001726	460	0.29	
	400 F. (205 C.)	22 500	0.004078	293	....	Bar failed, 300 hr.
	600 F. (315 C.)	1 300	0.000273	145	0.00	
	600 F. (315 C.)	2 500	0.001651	293	0.28	
	600 F. (315 C.)	3 400	0.003906	312	1.04	
	600 F. (315 C.)	4 300	0.008777	267	....	
	600 F. (315 C.)	5 000	0.017813	251	....	
No. 1-A	400 F. (205 C.)	15 000	0.000875	556	0.11	
	400 F. (205 C.)	20 000	0.010090	532	....	
	600 F. (315 C.)	2 500	0.002688	317	0.69	
	600 F. (315 C.)	5 000	0.042150	79	....	
No. 2	400 F. (205 C.)	5 000	0.000322	511	0.01	
	400 F. (205 C.)	10 000	0.000916	534	0.11	
	400 F. (205 C.)	15 000	0.026033	668	....	Extension at 506 hr., 0.010905 in. per inch.
	400 F. (205 C.)	20 000	0.012269	28	....	Bar failed, 28.5 hr.
	600 F. (315 C.)	1 300	0.000644	383	0.12	
	600 F. (315 C.)	1 900	0.001710	294	0.43	
	600 F. (315 C.)	2 500	0.003310	312	0.99	
	600 F. (315 C.)	3 400	0.019899	175	....	
	600 F. (315 C.)	5 000	0.0740	47	....	
No. 3	400 F. (205 C.)	5 000	0.000413	503	0.05	
	400 F. (205 C.)	7 500	0.000570	579	0.07	
	400 F. (205 C.)	10 000	0.000908	554	0.12	
	400 F. (205 C.)	12 500	0.002312	486	0.17	
	400 F. (205 C.)	15 000	0.006705	510	....	
	600 F. (315 C.)	1 300	0.000602	245	0.15	
	600 F. (315 C.)	1 900	0.001205	312	0.25	
	600 F. (315 C.)	2 500	0.004640	310	1.38	
	600 F. (315 C.)	3 400	0.020691	96	....	
No. 4	400 F. (205 C.)	2 500	0.000355	509	0.04	
	400 F. (205 C.)	5 000	0.000789	535	0.09	
	400 F. (205 C.)	7 500	0.001164	484	0.14	
	400 F. (205 C.)	10 000	0.004368	510	0.69	
	600 F. (315 C.)	1 300	0.000654	308	0.13	
	600 F. (315 C.)	1 900	0.001198	308	0.29	
	600 F. (315 C.)	2 500	0.003798	308	....	
	600 F. (315 C.)	3 400	0.015852	72	....	Bar failed, 75 hr.
No. 5	400 F. (205 C.)	5 000	0.000520	578	0.06	
	400 F. (205 C.)	7 500	0.000829	578	0.09	
	400 F. (205 C.)	10 000	0.001246	578	0.15	
	400 F. (205 C.)	15 000	0.011224	555	....	Extension at 508 hr., 0.008339 in. per inch.
	600 F. (315 C.)	1 300	0.000768	169	0.21	
	600 F. (315 C.)	2 500	0.016071	266	....	
No. 6	400 F. (205 C.)	7 500	0.000553	574	0.07	
	400 F. (205 C.)	10 000	0.001445	788	0.14	
	400 F. (205 C.)	15 000	0.016109	576	....	
	600 F. (315 C.)	1 300	0.000950	291	0.22	
	600 F. (315 C.)	1 900	0.003013	316	0.87	
	600 F. (315 C.)	2 500	0.017090	340	....	
No. 7	400 F. (205 C.)	2 500	0.000256	523	0.03	
	400 F. (205 C.)	5 000	0.000603	576	0.09	
	400 F. (205 C.)	7 500	0.000743	523	0.12	
	400 F. (205 C.)	12 500	0.005260	557	....	
	600 F. (315 C.)	1 300	0.001469	308	0.40	
	600 F. (315 C.)	1 900	0.004029	311	1.17	
	600 F. (315 C.)	2 500	0.025405	311	....	
No. 8	400 F. (205 C.)	2 500	0.001132	600	0.15	
	400 F. (205 C.)	5 000	0.003030	527	0.42	
	400 F. (205 C.)	10 000	0.013953	269	....	
	600 F. (315 C.)	1 300	0.003104	313	0.86	
	600 F. (315 C.)	1 900	0.007803	311	2.09	
	600 F. (315 C.)	2 500	0.018825	266	....	
	600 F. (315 C.)	3 400	0.0495	71	....	Bar failed, 72 hr.

window of the furnace. The extension of the specimens was determined by focusing the microscope on this area and measuring the distance between selected spots on the square impressions by means of the filar eyepiece. The extension of the specimens was read on one side only.

The apparatus meets the requirements of the A.S.T.M. Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 34 T).[1]

ALLOYS USED AND RESULTS OF TESTS

A list of the alloys used is given in Table I, with the chemical composition of each. The method of heat treatment is given in cases where it was known. Some of the alloys such as Nos. 1 and 2 have been used for a long period under exacting conditions at elevated temperatures. Others are later developments, while some have not as yet gained acceptance. The list includes most of the alloys used at elevated temperatures in American practice.

The results of short-time tension tests at atmospheric temperature, at 400 and 600 F. (205 and 315 C.) are given in Table II. The results given for alloys Nos. 1, 2, 3 and 4 were obtained on specimens from the same lot of metal as that used for creep tests in each case. As stated above, the specimens for short-time elevated-temperature tests received the same stabilizing treatment before testing that was given the specimens for creep tests. The results given for the other alloys were reported by the manufacturer as being typical of these alloys.

The results of long-time or creep tests are given in Table III and Figs. 3 to 8. Tests were run for 300 hr. at 600 F. (315 C.) and for 500 hr. at 400 F. (205 C.). The total extension over the entire testing period is reported in inches per inch. The extension during the secondary period is reported as creep rate per 1000 hr. or the period of time considered to be the service life of a military aircraft engine. The creep rate is not reported in cases where the specimen was rapidly extending toward the close of the testing period and would probably have failed in less than 1000 hr.

DISCUSSION

Marked differences were observed in the mechanical properties of the alloys as determined by short-time tests. The results given in Table II indicate that at room temperature the wrought alloys (Nos. 1-A, 5, 6 and 7) had much higher tensile strength and ductility than the cast alloys (Nos. 1, 2, 3, 4 and 8). In short-time tests at 400 F. (205 C.) the wrought alloys showed a greater drop in tensile strength than the cast alloys, though

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1223 (1934); also 1934 Book of A.S.T.M. Tentative Standards, p. 1147.

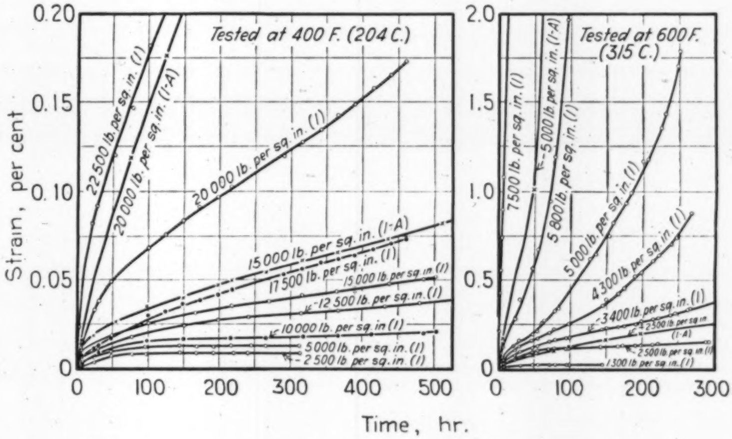


FIG. 3.—Creep Test Results for Alloys Nos. 1 and 1-A.

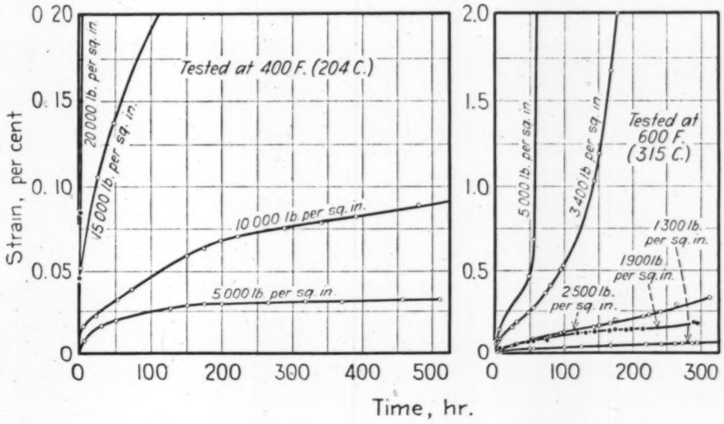


FIG. 4.—Creep Test Results for Alloy No. 2.

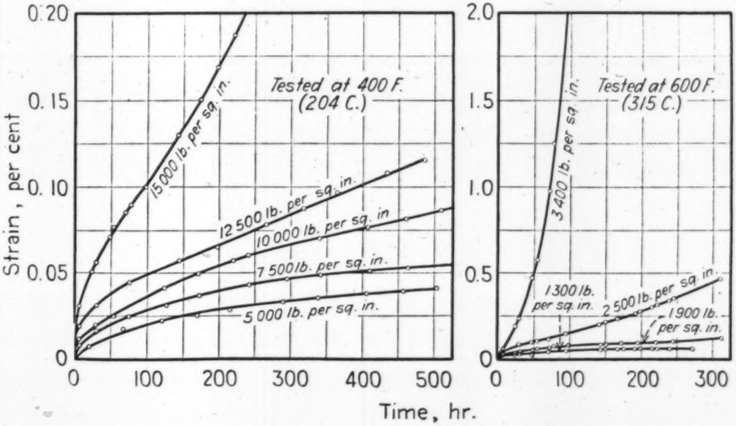


FIG. 5.—Creep Test Results for Alloy No. 3.

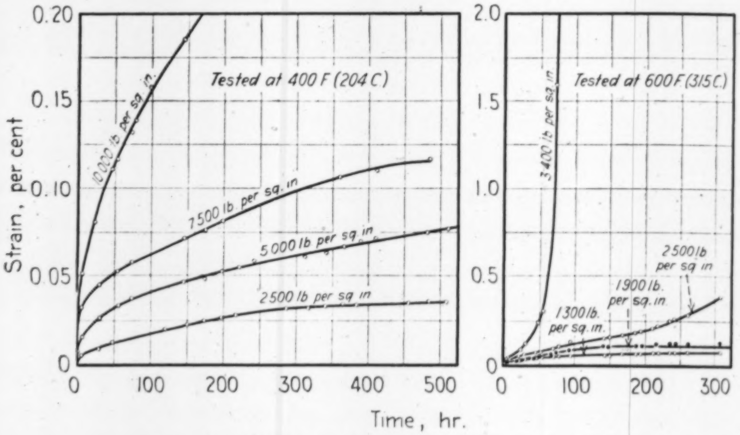


FIG. 6.—Creep Test Results for Alloy No. 4.

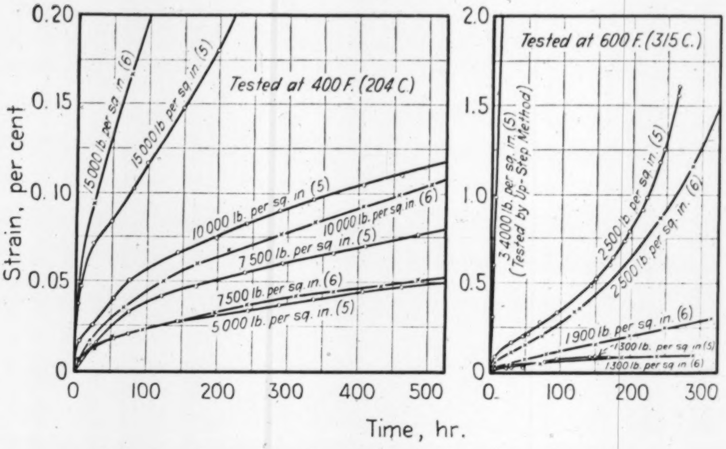


FIG. 7.—Creep Test Results for Alloys Nos. 5 and 6.

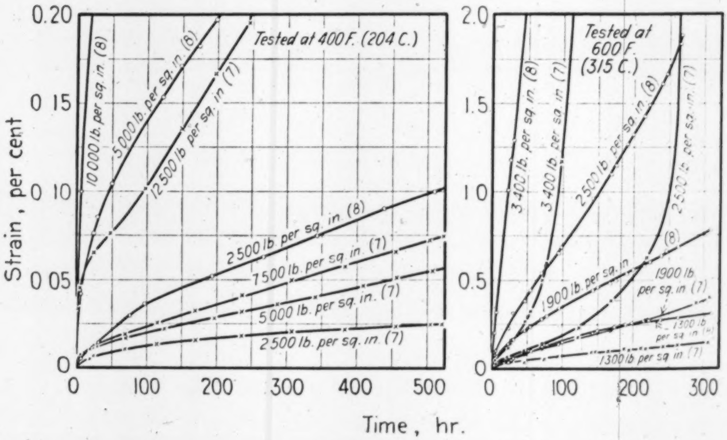


FIG. 8.—Creep Test Results for Alloys Nos. 7 and 8.

wrought alloys Nos. 1-A and 5 were stronger than the cast alloys. Wrought alloys Nos. 6 and 7 were heated for long periods before testing, so the results are not comparable. At 600 F. (315 C.) the strength of all alloys was greatly reduced as compared with the properties at room temperature. Cast alloy No. 1 (4 per cent copper, 2 per cent nickel, 1.5 per cent magnesium) had the highest tensile strength.

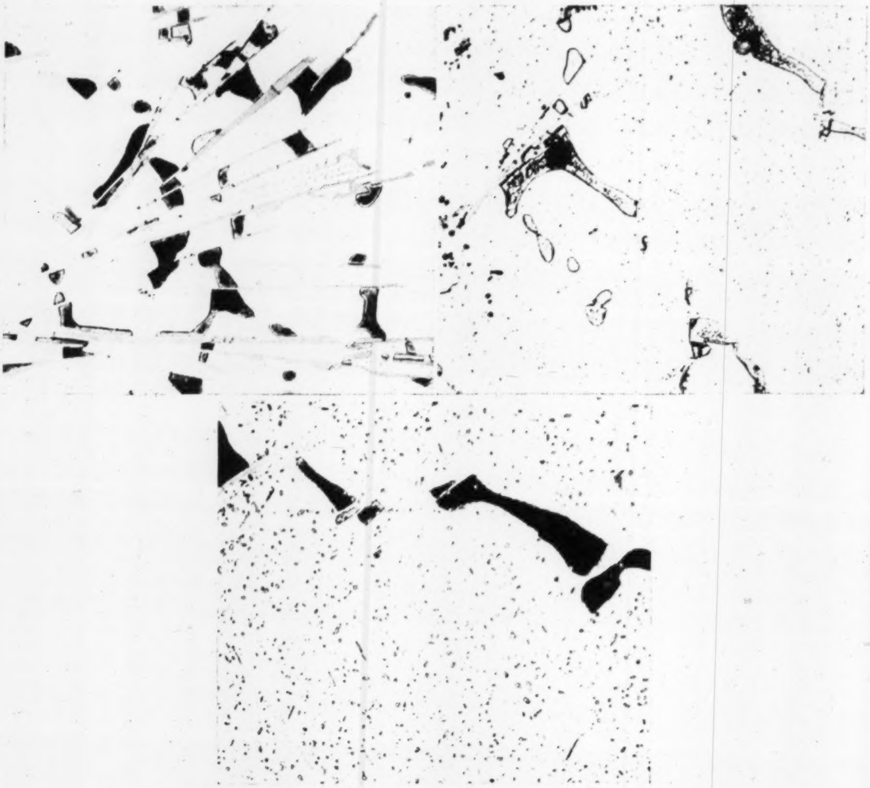
The creep rates of the alloys varied over a wide range. The rate of extension at 600 F. (315 C.) was much more rapid than at 400 F. (205 C.) as would be expected. Cast alloy No. 1 had the best creep characteristics of the alloys tested, both at 400 and at 600 F. (205 and 315 C.). Alloy No. 1-A had the same chemical composition as alloy No. 1 but was forged into the form of a bar. The forged alloy specimens had appreciably higher creep rates than the cast alloy in the tests run. Unfortunately only a few specimens of the forged alloy were available, but the results indicate that hot working with its consequent grain refinement may have a deleterious effect on the creep characteristics of aluminum alloys. The other alloys tested had higher creep rates than alloys Nos. 1 and 1-A, the cast alloys in general having better creep characteristics than the forged alloys, though a few exceptions may be noted. Cast alloy No. 8 (6 per cent magnesium, 1.5 per cent nickel, 1 per cent manganese) had the highest creep rate of the alloys tested.

TABLE IV.—HARDNESS OF ALLOYS AFTER CREEP TESTS

Alloy	Temperature of Test	Duration of Test Prior to Hardness Testing, hr.	Brinell Hardness, 500-kg. load
No. 1	400 F. (205 C.)	503	96
	600 F. (315 C.)	293	54
No. 1-A	400 F. (205 C.)	556	105
	600 F. (315 C.)	317	60
No. 2	400 F. (205 C.)	511	91
	600 F. (315 C.)	264	53
No. 3	400 F. (205 C.)	503	67
	600 F. (315 C.)	246	39
No. 4	400 F. (205 C.)	509	74
	600 F. (315 C.)	308	46
No. 5	400 F. (205 C.)	578	75
	600 F. (315 C.)	308	49
No. 6	400 F. (205 C.)	574	79
	600 F. (315 C.)	291	60
No. 7	400 F. (205 C.)	576	49
	600 F. (315 C.)	311	44
No. 8	400 F. (205 C.)	527	81
	600 F. (315 C.)	313	76

EFFECT OF CREEP TESTING ON THE ALLOYS

The hardness of the alloys was greatly reduced by prolonged exposure to testing temperatures as shown in Table IV. Hardness tests were made on the shoulder of the specimen as near the gage length as possible after the specimen had cooled to room temperature. This portion of the specimen had been heated to the testing temperature for the period of the test, but had not been excessively strained by the load applied. Forged alloy No. 1-A (4 per cent copper, 2 per cent nickel, 1.5 per cent magnesium) had the highest hardness of the alloys tested after 500 hr. heating at 400 F. (205 C.) while alloy No. 8 (6 per cent magnesium, 1.5 per cent nickel, 1 per cent manganese) which had the highest creep rate of the series, had the highest hardness after 300 hr. heating at 600 F. (315 C.).



(*a*) As heat treated.

(*b*) After creep testing for 500 hr. at 400 F. (205 C.).

(*c*) After creep testing for 300 hr. at 600 F. (315 C.).

FIG. 9.—Structure of Alloy No. 2 (× 500).

A number of specimens were examined under the microscope to determine the effect of heating on the structure of the alloys. The most notable structural change observed was the abundant precipitate produced by the long period of heating. Aluminum alloys low in manganese, when heat treated to produce maximum tensile properties, generally show no evidence of a precipitated constituent that can be detected by microscopic examination. The structure of alloy No. 2 (10 per cent copper, 1 per cent iron, 0.25 per cent magnesium) in the heat-treated condition may be considered typical (Fig. 9(*a*)). The solution treatment served to dissolve all of the soluble constituents which could go into solid solution under the conditions employed. The alloy after solution treatment consists of solid solution, excess soluble constituents and insoluble compounds. The aging treatment, while it greatly improves the mechanical properties of the alloy does not alter the microstructure. The particles of precipitate, if the aging treatments ordinarily applied actually cause precipitation, are submicroscopic. Exposure to a temperature of 400 F. (205 C.) for 500 hr. produces particles of precipitate which are readily detectable as shown in Fig. 9 (*b*), although 400 F. (205 C.) is in the aging range for aluminum alloys. A temperature of 600 F. (315 C.) is in the annealing range so a rapid growth of the particles of precipitate would be expected. Figure 9 (*c*) shows a specimen of alloy No. 2 which was heated to 600 F. (315 C.) for 300 hr.

COMPARISON OF LONG-TIME AND SHORT-TIME PROPERTIES

The mechanical properties of the alloys at room temperature apparently bear no relation to the creep characteristics at elevated temperatures. Some of the alloys such as alloy No. 7 (4 per cent copper, 2 per cent nickel, 0.5 per cent magnesium) had high strength at room temperature and relatively poor creep characteristics. Due to the progressive loss of strength which takes place when aluminum alloys are heated to elevated temperatures for long periods, it would not be expected that the creep characteristics could be deduced from the results of short-time tests at those temperatures. Both types of tests were run on cast alloys Nos. 1, 2, 3 and 4 under comparable conditions. When these alloys are listed in the order of their tensile strengths as determined by short-time tests at 400 F. (205 C.), alloy No. 1 has the highest tensile strength followed in order by alloys Nos. 2, 3 and 4. The results of creep tests indicate that the alloys fall in the same order. Alloy No. 8, however, which has a short-time tensile strength between alloys Nos. 3 and 4, has an extremely high creep rate. At 600 F. (315 C.) the positions of alloys Nos. 3 and 4 are reversed both in long-time and in short-time tests.

Insufficient data were available for the wrought alloys so the relationship between the cast and the wrought alloys could not be deduced.

CONCLUSIONS

Caution should be used in applying the results of creep tests to the design of airplane-engine parts due to the difference between test conditions and service conditions. In a creep test, both the load and the temperature are constant for the period of the test while in an airplane engine the parts are subjected to vibration, often to alternating tensile and compressive stresses and the temperature fluctuates according to operating conditions. The results of creep tests should be of value, however, in indicating the relative behavior of the various alloys at operating temperatures.

Some correlation has been observed between creep characteristics and service performance in the case of air-cooled cylinder heads for airplane engines. These parts are cast from alloys Nos. 1 and 2 which have good creep characteristics. They have proven satisfactory in service and few failures have been reported. Recently, cylinder heads of the same type have been cast from alloy No. 8 which has poor creep characteristics. Many of these cylinder heads have failed during block tests of the engine. Alloy No. 8 has low heat conductivity, so these cylinder heads operate at temperatures approximately 50 F. (28 C.) higher than cylinder heads cast from alloys Nos. 1 and 2. The higher operating temperature would tend to lower the mechanical properties of the alloy, but it is probable that the high creep rate is an important factor in the failure of the cylinder heads.

The same correlation has not been observed in the case of airplane engine pistons. These parts were formerly cast from alloys Nos. 1 and 2 but are now forged from alloys Nos. 5 and 7 which have poorer creep characteristics. The wrought alloys have given satisfactory service in spite of their high creep rates. As in the case of the cylinder heads, a difference in operating temperature is probably a contributing factor, because, due to changes in piston design, the wrought pistons run cooler than do the cast pistons.

Acknowledgment:

The author wishes gratefully to acknowledge the aid of the following individuals and organizations: H. W. Gillett and H. C. Cross of the Battelle Memorial Institute for their kind suggestions and aid in the construction of the apparatus. T. D. Stay and G. D. Welty of the Aluminum Company of America who furnished the wrought alloys and one of the cast alloys used. R. L. Templin of the same organization who kindly furnished results of mechanical tests of these alloys. J. B. Johnson, Chief of the Material Branch at Wright Field, under whose supervision the investigation was conducted and the members of the staff at Wright Field, particularly C. F. McMahon who aided in the work.

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(7) F. M. Howell and D. A. Paul, "The Properties of Aluminum and Two of Its Alloys at Elevated Temperatures," *Metals and Alloys*, Vol. 5, August, 1934, p. 176.

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DISCUSSION

MR. F. M. HOWELL[1] (*presented in written form*).—The author has presented some very interesting data upon the behavior of some of the aluminum alloys most generally used at elevated temperatures. In the Aluminum Research Laboratories we have made tension tests at elevated temperatures on most of these alloys and creep tests on several of them. In general, our results are in agreement with those obtained at Wright Field.

It may be of some interest to describe the method we follow in determining the stabilizing treatment used for our creep specimens. In making our tension tests at elevated temperatures it is our practice to place a number of specimens in a furnace operating at the temperature at which the tests are to be made. Specimens are then removed at intervals, placed in the furnace in which the high-temperature tests are made and when they have been thoroughly reheated to this temperature the tension test is made. This procedure is continued with successive specimens until no further change in tensile properties is observed. In some cases, depending upon the alloy and the temperature, this requires a relatively short period of time, that is, 10 days or two weeks, and in other cases requires two years or more. Specimens that are to be used for creep tests are heated at the testing temperature for the length of time indicated by the tension tests as necessary for complete stabilization.

In the case of materials such as those tested by the author our stabilizing times are very much greater than those used at Wright Field. The stabilizing treatments used by the author, however, seem quite adequate for the particular problem at hand.

MR. R. R. KENNEDY.[2]—I want to thank Mr. Howell for his contribution. We recognized the fact that we were not completely stabilizing our alloys and mentioned in the paper that the results of the test on the completely stabilized alloys would be desirable; therefore I think the forthcoming work of Mr. Howell will be a distinct contribution to the literature. In our problem, that of aluminum alloys in aircraft, where the total life of the mechanism is only about a thousand flying hours, we did not think it would justify completely stabilizing the alloys before testing.

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[1] Engineer of Tests, Research Laboratories, Aluminum Company of America, New Kensington, Pa.
[2] Associate Metallurgist, U. S. Army Air Corps, Wright Field, Dayton, Ohio.

CONTINUOUS-FLOW CORROSION TESTS OF STEEL PIPE[1]

By H. S. Rawdon[2] and L. J. Waldron[2]

Synopsis

The shortcomings of ordinary laboratory corrosion tests, particularly for predicting what may be expected to occur in service, are well recognized. The limitations imposed by the small size of the usual specimen, the relatively short duration of the test and the failure to simulate the essentials of the conditions expected in service are often very serious ones. Exposure tests on the other hand are not always practicable, and the possibility of developing a testing procedure substantially free from the really serious shortcomings of ordinary laboratory corrosion tests remains still a subject of live interest. The method described here has been used with ferrous pipe material and consists essentially in circulating water of known characteristics through a system built up of sections of the pipe to be tested. A number of specimens approximately 5 in. long, after being cleaned and weighed, were assembled, with intermediate hard rubber spacing rings, to form a vertical water-tight column. After being subjected to water flowing at a controlled rate for the desired period, the columns are disassembled and the specimens properly cleaned, weighed and the corrosion rate determined.

Results obtained on 2-in. pipe of two open-hearth steels showed: (*a*) distribution of corrosion varies decidedly with the degree of turbulence in the flow; (*b*) corrosion of specimens varies with position in the column, the rate being a maximum at the inlet end with a progressive decrease along the column, which effect can be largely overcome by increasing the rate of flow; (*c*) pipe used successively from one series of tests to another, the specimens being cleaned at the end of each run, showed an apparent increase in corrosion rate with increased use, attributable to roughening of the surface—a factor which is not usually taken into consideration; (*d*) the distribution of corrosion is intimately related to scale deposition accompanying corrosion.

Introduction

The shortcomings of ordinary laboratory corrosion tests, particularly for use as a basis for predicting what may be expected to occur in service, are well recognized. Such objections are not necessarily to be regarded as adverse criticism of the accuracy and care with which the tests are made. The limitations imposed by the small size of the usual specimen, the relatively short duration of the test and the failure of the conditions under which the tests are carried out to simulate the essentials of those expected in service are often very serious ones. Evaluation of any material by actual service is, of course, ideal but usually this is not practicable. Whether or not such an evaluation can ever successfully be made by any test, short of service, is a matter of conflicting opinion. However, the possibility of developing a testing procedure which is substantially free from the really serious objections to which the ordinary corrosion test is subject is of decided interest. The present paper deals with a testing method used at the National Bureau of Standards with some measure of success as applied to ferrous material in the form of pipe. The test may perhaps be more properly termed a short-time service test rather than an extended laboratory test.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Chief, Division of Metallurgy, and Assistant Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

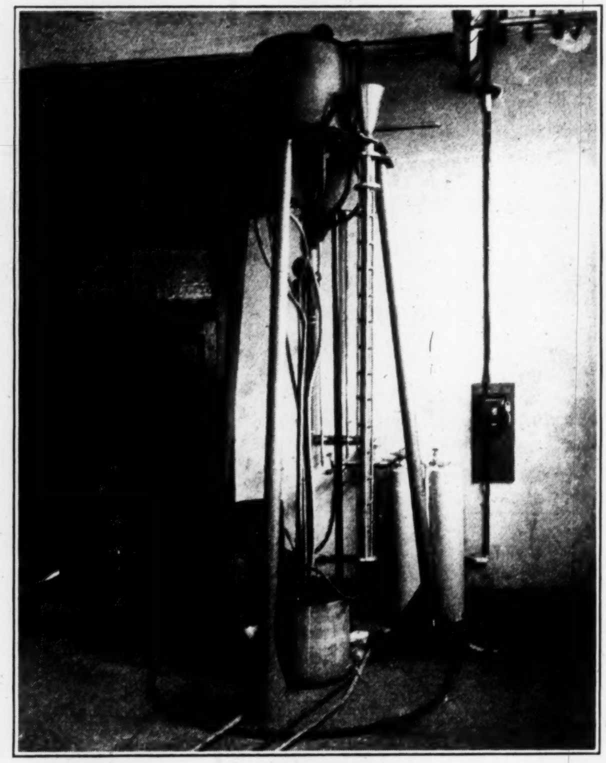

FIG. 1.—Apparatus for the Continuous-Flow Corrosion Tests of Pipe.
One column of specimens is shown.
The over-all height of the apparatus is approximately 13 ft.

APPARATUS AND METHOD

The method consists essentially in circulating water of known characteristics through a system built up of sections of the pipe to be tested. The sections are large enough to be representative of the pipe and still not beyond the limits of laboratory use. The apparatus used is shown in Fig. 1

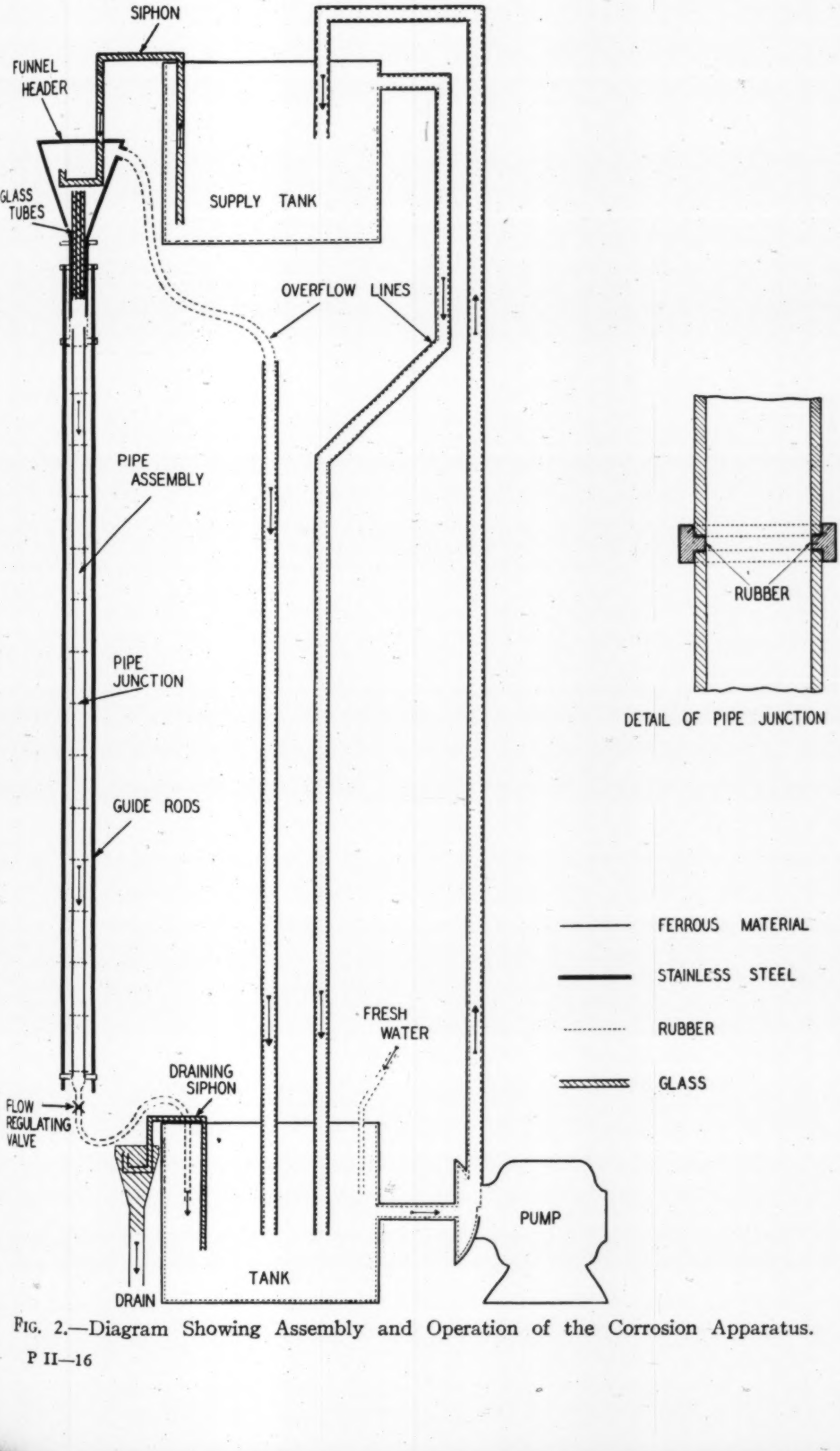
SIPHON
FUNNEL HEADER
GLASS TUBES
SUPPLY TANK
OVERFLOW LINES
PIPE ASSEMBLY
PIPE JUNCTION
GUIDE RODS
RUBBER
DETAIL OF PIPE JUNCTION
FERROUS MATERIAL
STAINLESS STEEL
RUBBER
GLASS
FRESH WATER
DRAINING SIPHON
FLOW REGULATING VALVE
PUMP
TANK
DRAIN

FIG. 2.—Diagram Showing Assembly and Operation of the Corrosion Apparatus.

with an assembled column of test specimens in place. The construction and working of the apparatus are shown diagrammatically in Fig. 2.

In the work so far, the test samples consisted of sections of pipe approximately 5½ in. in length having an inside area of approximately 35 sq. in. (2.3 sq. dm.). A number of specimens, usually 14, were assembled into a column with a hard-rubber washer separating adjacent specimens to prevent electrical contact. Through this column, which was supported in a vertical position and held together and maintained in a water-tight condition by suitable external tie rods, water was passed continuously, the quantity being sufficient to fill completely the assembled pipe column. The object sought was to expose continuously, as in service, the different samples, in as nearly an identical manner as possible, to water of known pH value, oxygen concentration and velocity of flow.

The rate of flow of water through the column was regulated by adjusting the orifice at the lower (outlet) end of the column. The water in the supply tanks was recirculated through the column of pipe specimens. The capacity of the system was approximately as follows: lower tank, 30 gal.; upper tank, 25 gal.; each column, 1½ gal. A small amount was allowed to drain away from the lower tank and a corresponding amount of fresh water was continuously added to the system, the quantity being such as to be equivalent to a complete renewal of the water in the system each 24 hr.

For much of the work, the water used was taken directly from the tap and hence was representative of the Washington water supply. The pH value of this water, as determined on samples taken from the lower tank (Fig. 2), ranged between 7.6 and 7.9. In the later runs, the tap water before being passed through the system was softened by means of a commercial softener, utilizing the base-exchange principle whereby the calcium and magnesium salts in water are converted to sodium salts. In addition some CO_2 was passed into the water to increase its corrosive qualities, in amounts sufficient to lower the pH value to 6.0. The colorimetric method (Taylor Slide Comparator) was used for all pH determinations. The concentration of dissolved oxygen lay between 5 and 7 cu. cm. per liter and the average temperature was 68 F. (20 C.).

Throughout the work, the oxygen content of the water was measured at frequent intervals by the method generally referred to as the Winkler method.[1]

MATERIAL

Open-hearth steel pipe having an internal diameter of 2 in. and a wall thickness of $\frac{3}{16}$ in., manufactured by the electric-resistance or flash-welding process, was used. The inner line of the weld was planished off so as to remove the slightly projecting "flash." The compositions of the materials used were as follows:

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[1] Proposed Method for Determination of Dissolved Oxygen in Boiler Feed Water, *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part I, p. 178 (1929).

	STEEL K	STEEL F
Carbon, per cent	0.10	0.11
Manganese, per cent	0.45	0.43
Phosphorus, per cent	0.014	0.012
Sulfur, per cent	0.027	0.029
Silicon, per cent	0.005	0.005
Copper, per cent	0.30	0.12

Material K was used in most of the tests reported here. Material F, secured with the hope that it would be free from copper, was used in the later tests.

All the specimens at the outset were cleaned free from mill scale by the following procedure: (*a*) scrubbed in 10 per cent NaOH solution, to remove grease, etc.; (*b*) rinsed in water; (*c*) pickled in hot 10 per cent HCl solution containing a commercial inhibitor; (*d*) washed in hot running water; (*e*) dried. Wire brushing was used when necessary. The specimens were weighed to 0.005 g., the weight of the specimens being 700 to 800 g., approximately. At the close of a run, the specimens were cleaned free from corrosion products, before being reweighed, by the following procedure: (*a*) brushed with soft bristle brush under running water; (*b*) pickled electrolytically by being made the cathode (25 amp. per sq. ft.) in 10 per cent HCl solution (with an inhibitor); (*c*) brushed with wire brush; (*d*) washed in hot running water; (*e*) dried. The average corrosion loss was calculated from the loss in weight, the nominal area exposed to the water and the corrosion period as milligrams per square decimeter per day (24 hr.). It should be emphasized that the corrosion rate determined in this manner expresses only the average behavior of the material; cognizance is not taken of any tendency toward localized attack.

The various sets of specimens were used continuously from one run to another; that is, after the specimens were cleaned and weighed at the completion of a run, they were used in the succeeding run, no attempt being made to remove any slight roughening which had occurred as a result of corrosion. In this respect the test simulated service conditions. The stopping of the tests at intervals for the removal and cleaning of the specimens in order to determine their corrosion losses, of course, does not. Prior to the last run, all specimens were machined on their inner surface to remove all evidence of previous corrosion. A roughing cut was first taken and the finishing done in a precision lathe; a finely machined surface was produced.

TEST RESULTS

Examination of specimens used in preliminary runs indicated the distribution of corrosion to be influenced decidedly by the swirling flow of the water in the pipes, the preferential attack of specimens at the inlet end

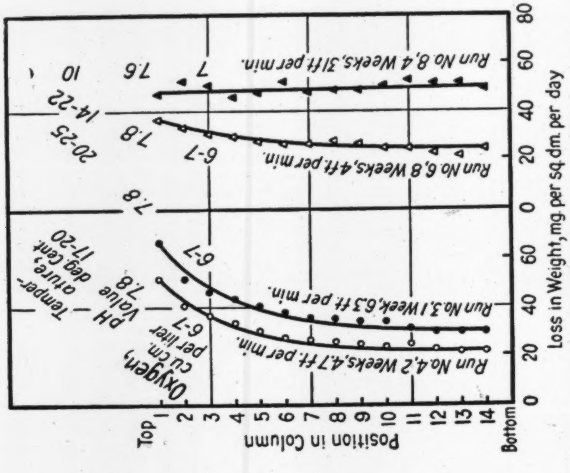

FIG. 3.—Results With Material K Illustrating Effect of Flow Rate and Duration on the Distribution of Corrosion Within Each Column of Specimens.

Each curve represents a column of 14 specimens of material K.

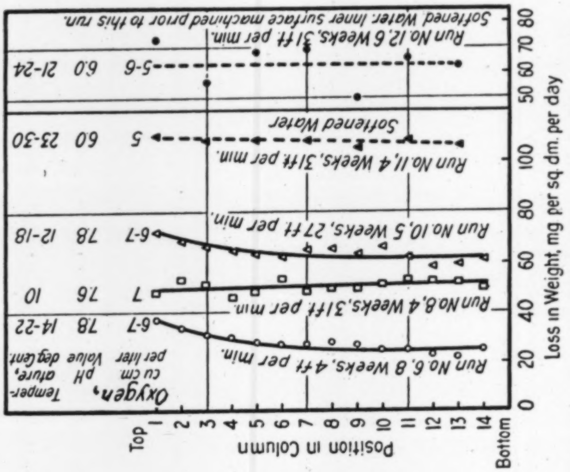

FIG. 4.—Results With Material K Illustrating *Apparent* Increase in Corrosion Rate in Successive Runs on the Same Specimens and the Difference of Corrosion in Tap Water and in Softened Water.

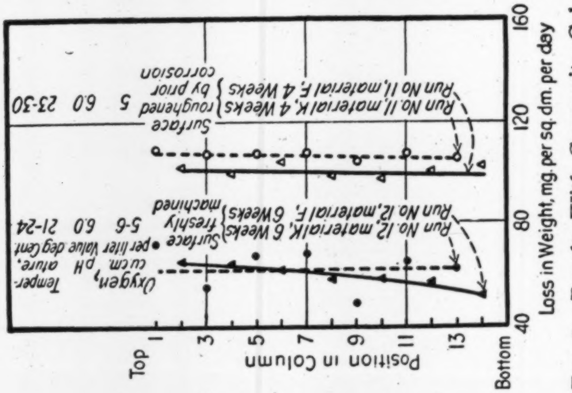

FIG. 5.—Results With Composite Columns of Alternating Specimens of Materials K and F in Softened Water, Flow Rate 31 ft. per min.

being even more pronounced than is shown in Fig. 3. The corroded surface showed a spiral pattern indicative of the swirling flow of the water. By inserting a bundle of parallel glass tubes ($\frac{5}{16}$-in. inside diameter) in the lower end of the feeding head and by smoothing the rubber washers and junction of adjacent specimens, conditions were greatly improved. All of the results shown in the figures were obtained after these changes had been made and a fairly straight-line flow of water had been obtained.

The results obtained in several series of runs are summarized graphically in Figs. 3 to 5, inclusive. The sloping curves (Fig. 3), indicating a maximum corrosion rate at the inlet, with progressively lower corrosion rates for specimens constituting the upper half of the column was quite characteristic for slow rates of water flow. By increasing the corrosion period, this difference in corrosion rate tended to decrease. A considerably higher flow rate was more effective, however, in producing fairly uniform corrosion rates throughout the column.

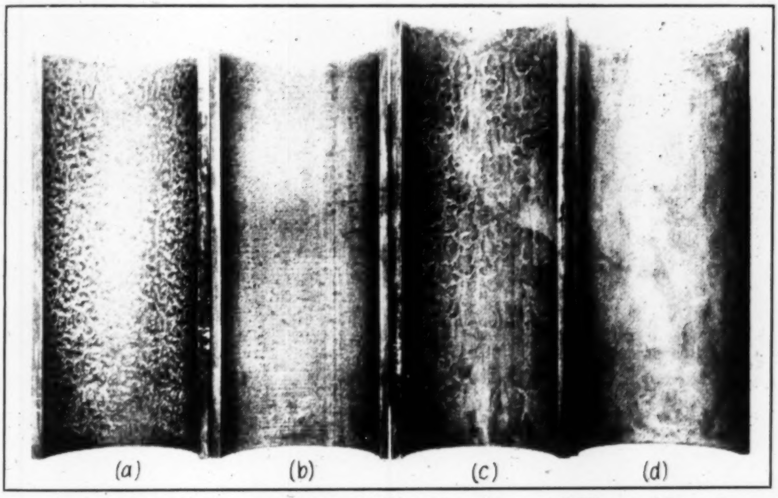

FIG. 6.—Appearance of Inner Surface of Pipe Specimens of Fig. 5 After Corrosion.

(*a*) Material K, used successively for 5 runs (14 weeks total).
(*b*) Material K, machined before run No. 12 in softened water.
(*c*) Material F, one run in tap water.
(*d*) Material F, machined before run No. 12 in softened water.

The curves in Fig. 4 show the apparent increase in average corrosion rate (loss of weight, milligrams per square decimeter per day) of the same specimens used in successive runs under the same condition. The change from one type of water to another of different characteristics undoubtedly does cause a change in corrosion rate. Other changes in corrosion rate are probably only *apparent* and are the result of progressively increasing surface roughening. This was apparent when the tests were made on specimens from which the previously corroded inner surface had been machined off.

In Fig. 5 are given the results obtained for the two steels corroded under comparable conditions, the two materials forming alternate specimens in the same column. Differences of the magnitude shown are apparently of no real significance.

The appearance of the corroded inner surface of the pipe specimens shown in Fig. 6 is typical of the specimens corroded in the manner described. All specimens used consecutively in a series of runs in tap water had the mottled appearance shown. Apparently this is characteristic of the action of a scale-forming water. In these tests this pattern persisted even though the final corrosion of the specimens was carried out in the more corrosive softened water. It did not form, however, on the same materials, freshly machined, when corroded in the softened water. The deposition of scale simultaneously with corrosion apparently has a marked effect on the distribution of corrosion. The reticulations or network (lighter areas, Fig. 6) were in relief and were not so severely corroded as the darker intervening patches. That the mottled surface condition is closely associated with scale deposition was readily demonstrated when the surface was cleaned by pickling. Although the mottled appearance persisted from one run to another, the surface being chemically cleaned between runs, there is no assurance that the pattern in one run was maintained essentially unchanged in the succeeding run. In spite of this factor, however, the average corrosion losses of the specimens constituting a column were surprisingly uniform in most cases if the flow rate was not too low.

In accordance with the recommendations of the Society,[1] the data were studied by the method of statistical analysis. This study of the various sets of data which could be represented fairly well by straight lines, Figs. 3 to 5, gave results indicating "controlled conditions." The proper interpretation to be given to this, however, in view of the visual evidence of non-uniformity of the corrosion attack, is a matter of grave uncertainty. For those sets of data showing wide scatter or a marked variation of corrosion rate along the length of the column, statistical analysis, although applied, could not be expected to throw much useful additional light on the subject.

SUMMARY

The useful aspects of the work may be summarized as follows:

1. The distribution of corrosion within a water pipe was found to be affected by the degree of turbulence in the flow. For most satisfactory results, straight-line flow was necessary.

2. The corrosion rate within a column assembled out of specimens cut from the same pipe varied decidedly according to conditions of flow. The

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[1] Manual on Presentation of Data, sponsored by Committee E-1 on Methods on Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 451 (1933); also available as separate publication.

maximum rate was at the inlet and progressively decreased along the length of the column. This difference in corrosion rate, which was most pronounced with low flow rates, tended to become less as the corrosion period was extended and was practically eliminated by using a considerably higher rate of flow.

3. Pipe used successively in a series of corrosion tests, the specimens being cleaned free of corrosion products at the end of each run, however, showed an increasing corrosion rate in successive runs. This was only *apparent*, however, and evidently should be attributed to the increase in surface area resulting from the progressive roughening by corrosion, a factor which cannot be taken into account in calculating the rate.

4. The distribution of the corrosion of pipe in ordinary tap water was affected by the scale deposition occurring simultaneously with corrosion. A mottled appearance resulted, certain areas constituting a general network showed practically no corrosive attack. However, the "over-all" corrosion rates of specimens corroded under such conditions were very uniform. In a more corrosive (softened) water the scatter in the results was much more pronounced.

Acknowledgment.—The laboratory work on which the paper is based was carried out by E. J. Ash and F. Sillers, Jr., Research Associates, National Research Council.

DISCUSSION

Mr. R. F. Passano.[1]—I was struck by the difference in the rate of corrosion observed on specimens exposed at the top and at the bottom of the column and the fact that the difference between specimens in the column decreased as the time of test and velocity of flow increased. It would appear to me that some of the difference might be due to oxygen being consumed by the specimens at the top of the column. Thus, the ones at the bottom of the column were actually subjected to water at a lower oxygen concentration than those at the top. The same explanation would serve as well in the case of an increase in velocity: the faster the water moves, the more chance there is of oxygen getting to the bottom of the column.

Mr. Rawdon referred to an apparent increase in the rate of corrosion as the specimens were used over and over again. I have compiled in the accompanying Table I the corrosion rate at the bottom of the column of specimens. I estimated these values from Figs. 3, 4, and 5 of the paper, which I presume are the data on which Mr. Rawdon's summary was based.

The only opportunity one has to compare the effect of successive runs at constant velocity is at approximately 5 ft. per minute in tap water. The series of rates 30, 21, and 24 mg. per sq. dm. per day so far as I can tell does not show any systematic difference attributable to the number of times the specimens have been used before. It seems to me that at *constant velocity* the rate of corrosion does not change as the specimens are used over and over again.

There is a reference in the paper to an "increase in scatter" when the corroding medium is soft water. The water which the authors call softened tap water was not only softened but made slightly acid with CO_2. If we compare run No. 8 and run No. 11, we find that the rate of corrosion in the first of these runs was 50 mg. per sq. dm. per day, and the rate of corrosion in the second of these runs was 105 mg. per sq. dm. As the data are presented in the paper, I do not see that there is a greater scatter of observations in run No. 11 than in run No. 8.

The data obtained in my laboratory at various times, some of which have been reported to the Society, indicate that as the loss of weight increases one should expect an increase in the standard deviation of the observations. So far as I have been able to determine, it seems to be almost a general property of numbers: the larger the magnitude of the thing measured in a particular system, the greater is the standard deviation of individuals.

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[1] Research Engineer, Research Laboratories, The American Rolling Mill Co., Middletown, Ohio.

Actually, the increase in scatter which the authors observed, and attributed to the softened water, was obtained on specimens which had been machined. I believe that the increase in scatter is a question of preparation of the surface rather than a question of the water used. The increase in scatter could just as well have been obtained on machined specimens exposed to tap water as machined specimens exposed to softened water.

Most investigators have reported a decrease in rate of corrosion as time increases. These reports have been based on specimens from which the rust has never been cleaned off. If one were going to make loss of weight measurements at one, two, five, and ten weeks, for example, one would start four sets of specimens. The first would be removed at the end of one week, the second at the end of two weeks, the third at the end of five weeks, and the fourth at the end of ten weeks. The decrease in rate of corrosion which has been observed on specimens so exposed has been due to an accumulation of corrosion products. The retardation brought about by this accumulation of corrosion products cannot be observed on a specimen which is cleaned at regular intervals. It is possible that specimens that are used over and over again may be roughened by the attack to such an extent that the area being corroded is greater than it was originally, but this effect is, I believe, small in comparison to irregularities due to incomplete attack of the specimen.

TABLE I.—CORROSION RATE AT BOTTOM OF COLUMN OF SPECIMENS.

Time, weeks	5 ft. per min.	25 ft. per min.	30 ft. per min.
WASHINGTON TAP WATER (pH 7.5)			
1	No. 3, 30 mg. per sq. dm.[a]	...	...
2	No. 4, 21 mg. per sq. dm.[a]	...	...
4	...	...	No. 8, 50 mg. per sq. dm.
5	...	No. 10, 61 mg. per sq. dm.[a]	...
8	No. 6, 24 mg. per sq. dm.[a]	...	...
SOFTENED TAP WATER + CO_2 (pH 6)			
4	...	...	No. 11, 105 mg. per sq. dm.
6	...	...	No. 12, 60 mg. per sq. dm. (machined)

[a] Columns of specimens that show higher rates at the top than at the bottom.

MR. F. N. SPELLER.[1]—This test should be classed as a pilot test and has many advantages in making preliminary evaluation of metals in tubular form. The uniformity of corrosion at a velocity of 30 ft. per min. between the top and the bottom is quite striking; apparently that should be the minimum velocity used. It is not a high velocity in water pipe. The final test, of course, should be made in service, but this test keeps the velocity and oxygen concentration uniform and eliminates any contact effects, and for preliminary work would have considerable advantage in testing the effect of small variations in composition. I cannot see any advantage in carrying a test of this kind to conclusion, because it leaves out such factors as variations in velocity, temperature, contact effect, and other factors that influence the results in service. It has been our custom to make a test very similar to this in actual service by installing different ferrous pipe together in the same line in order to get the effect of variations in velocity and temperature which have much to do with the ultimate results, especially the distribution of corrosion. Most users of metals are interested in the localization and depth of corrosion.

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[1] Director, Department of Metallurgy and Research, National Tube Co., Pittsburgh, Pa.

I should like to call attention again to the desirability of establishing a uniform method of expressing corrosion results. Three or four systems are in use at present; most of them mean very little except when they indicate the relative degree of corrosion. There is considerable advantage in expressing the average and maximum penetration in inches or mils per year or per month. This system gives most readers immediately a visual idea of the extent of the damage. I would again recommend the establishment of corrosion units for the consideration of the National Bureau of Standards. At least two different units of corrosion losses are now used in the Bureau itself.

MR. PASSANO.—Mr. Speller has raised the question of establishing a uniform practice for reporting corrosion results. His suggestion apparently is to report a corrosion rate, specifically penetration in inches (or mils) per month. I have something to say on this subject which I believe is pertinent.

The Society's Committee A-5 on Corrosion of Iron and Steel exposed a series of No. 22 and No. 16 gage ferrous sheets about 1917, and these were observed periodically for perforation. The time required to perforate No. 16 gage sheets of a given analysis was, in general, more than twice the time required to perforate No. 22 gage sheets of the same analysis.[1] This observation has since been satisfactorily explained by the knowledge that corrosion-time curves for this sort of exposure are not linear. If one divided the nominal thickness of No. 22 gage (0.03125 in.) by the time required for perforation, one would have a figure in the unit "inches penetration per month," but this rate would be different from that obtained by dividing the nominal thickness of No. 16 gage (0.0625 in.) by the time required to perforate it.

It is true that the selection of the unit "time required for perforation" was in some respects unfortunate. No data became available until the sheets perforated, and at some locations it was necessary to abandon the experiments before extensive data were available. It is natural, therefore, that some other method should be substituted for measuring the progress of corrosion. Loss of weight at definite intervals has been substituted by many investigators for time of perforation. It has the advantage that data are available as a result of each inspection.

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[1] R. F. Passano, "The Harmony of Outdoor Weathering Tests," Symposium on the Outdoor Weathering of Metals and Metallic Coatings, Washington Regional Meeting, Am. Soc. Testing Mats., p. 28 (1934); also discussion of same by J. C. Hudson, p. 45. Symposium available as separate publication.

So far as I know, there are no data available that allow one to correlate loss of weight with time required for perforation. Atmospheric corrosion tests with which I am familiar have shown, in a specific location, that copper-bearing steel required two or three times the exposure that similar sheets of open-hearth steel did to develop perforation; yet, loss of weight measurements made after six months, one year, two years, and four years, have shown in the same locality that copper-bearing steel loses approximately 85 per cent of the weight lost by ordinary steel.[1] Under such circumstances, it will be seen that the merits of the two materials when expressed in inches penetration per month would be one thing when based on time of perforation, and a quite different thing when based on loss of weight.

It is my recommendation that when corrosion data are available as a result of time required for perforation, they should be so reported. If they have been obtained as loss of weight, they should be reported as loss of weight per test specimen; the test specimen may need description too. Where measurements have been made on material of varying thickness or at a series of times, a corrosion-time curve should be considered as necessary information. The use of rates of corrosion should be restricted to those corrosion-time curves that are linear.

I have just indicated that it would be my recommendation to report loss of weight per specimen. It has not heretofore been considered bad practice to translate data obtained on 4 by 6, 12 by 12, or 12 by 24-in. specimens to "grams per 100 sq. cm." In this particular practice, one assumes that loss of weight is a random function of surface. In atmospheric investigations, I have good reason to believe that loss of weight is not a random function of surface. The loss of weight from one element of surface is correlated with that from other elements of surface. This is not astonishing when one recognizes that the drainage on 4 by 6-in. specimens, for example, is different from that on 12 by 24-in. specimens. This notion was observed first qualitatively by Mr. Taylerson and me at the time the tests of Committee A-5 at Fort Sheridan were abandoned. We thought that short sheets of a given analysis were in better condition than longer sheets of the same analysis.

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[1] *Loc. cit.*

The data given in the present paper by Messrs. Rawdon and Waldron are an excellent example of the fact that loss of weight is not a random function of surface. Suppose we use their run No. 4 as an example. There were 14 specimens in the column. The specimen at the top of the column showed a rate of 52 mg. per sq. dm. per day, and the one at the bottom showed a rate of 21 mg. per sq. dm. per day. If the column had been one nipple of pipe instead of 14 small nipples, the difference of rate observed would probably have been present on the single specimen, would not have been observed, and the rate for the single specimen might have been about 30 mg. per sq. dm. per day. We might even assume that the rate of corrosion on a single specimen (a 5½ in. length of pipe) is not uniform from one end to the other. Be that as it may, my point is that the loss from an element of pipe depends on the number of similar elements of pipe above it in the column.[1]

Corrosion-time relationships are in many respects similar to stress-strain curves obtained from tension tests. It has been the habit to abbreviate stress-strain curves into a series of values: yield strength, ultimate strength, and percentage elongation on the original gage length. When the material shows no definite yield point, the stress at an arbitrary amount of deformation is given as a substitute along with the ultimate strength and elongation.

I have said that corrosion-time relationships are similar to stress-strain curves of the sort which I have just described briefly. There are few persons who would concede that the information given by a stress-strain curve is conveyed by the modulus of elasticity which is analogous to rate of corrosion. Elongation corresponds to time in the corrosion process and ultimate strength to life or a point beyond which corrosion does not progress. Life or the point beyond which corrosion does not progress are significant things, I believe. I dislike arbitrary standardization that results in the suppression of valuable information and stifles progress.

I am sure that it would require considerable investigation before one could accomplish intelligent standardization of corrosion units in light of the facts reviewed in this discussion: namely, (1) corrosion-time relationships are often curved,[2] (2) a rate of corrosion—whether inches of penetration per month, or grams per square decimeter per day—does not adequately convey the information contained in a corrosion-time curve which is not linear, (3) loss of weight and time of perforation are probably not related linearly, and (4) loss of weight is not a random function of surface.

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[1] Discussion by R. F. Passano of paper by L. J. Waldron and E. C. Groesbeck, "Observations on Effect of Surface Finish on the Initial Corrosion of Steel Under Water," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 132 (1934).

[2] R. F. Passano, "The Corrosion-Time Relationship of Iron," *Industrial and Engineering Chemistry*, November, 1933, p. 1247.

The Society's Committee on Interpretation and Presentation of Data has recognized that the essential information in a series of observations cannot be conveyed by one function of the data. It recommends as a minimum the presentation of the conditions under which the observations were made, the average, the standard deviation, and the number of observations. In any proposed standardization, these considerations should be borne in mind, as well as the specific ones pertaining to this particular field cited above.

MR. H. E. SMITH.[1]—Mr. Passano's remarks have a bearing on quite an important detail in either laboratory or field tests. If the specimens are either vertical or at an angle, the corrosion is usually much greater on the lower end. In our black sheet field tests, all, or nearly all, fail within about a foot of the lower end and I think those who have made dipping tests in the laboratory have observed similar action. In the Bureau of Standards machine, the pieces are turned over every few days to overcome the difficulty mentioned. If something is not done to overcome that tendency, the results will be quite different from what they would be if the corrosion were uniform.

MR. H. S. RAWDON.[2]—On account of space limitations in publication, it was possible only to give results of representative test runs; all the data that had been obtained could not be given. For this reason some of the summarizing statements may not appear as conclusive as might be desired. Mr. Passano's comments on the statement concerning the apparent increase in corrosion rate with progressive roughening of the surface illustrate this point. It is true, as Mr. Passano pointed out, that this effect may be small with specimens exposed to tap water flowing at a low rate, 5 ft. per minute. Under conditions where scale is deposited from the water simultaneously with corrosion, this is not unexpected. However, in the nonscaling softened water of relatively low pH value the magnitude of the effect was quite different. The results shown in Fig. 5 are representative. Under the conditions obtaining in these runs, the corrosion rate of specimens roughened by previous corrosion was nearly 100 per cent greater than the rate observed on the same specimens after they had been given a smooth finish.

It is questionable whether the observed higher corrosion rate of specimens at top (inlet end) of the column as compared with that of specimens at the bottom (outlet end) of the column can be attributed solely to differences in oxygen supply of the water as it reaches these specimens. Attempts were made by analyzing, for oxygen, specimens of water taken simultaneously from top and bottom of the column to determine whether or not the assumption concerning the role of oxygen in this was justified.

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[1] Materials Engineer, White Plains, N. Y.

[2] Chief, Division of Metallurgy, National Bureau of Standards, Washington, D. C.

The results were inconclusive, however. The difference in corrosion rate was much more pronounced in the early experiments, before attempts were made to reduce swirling in the water by the introduction of the bundle of glass tubes.

The question raised by Mr. Speller of units for expressing results of corrosion studies is an important one. Mr. Passano's remarks are representative of the views held by many workers in this field that a single method of expressing such results is not advisable. In fact, one of the committees of the Society, Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, after a study of the matter has recommended to other committees of the Society and to research workers in general the use of two sets of units: (1) milligrams loss per square decimeter per day, and (2) inches penetration per year.[1] In a *great* many cases in which marked pitting of the metal does not occur, number 2 above is derived arithmetically from data obtained by number 1. When the purpose is obviously the comparison of closely related materials exposed to the same corrosive influences, as in the work under discussion, this arithmetical transformation would hardly seem necessary. However, whenever localized corrosion by pitting occurs on any materials, by all means the results of direct measurements of this should be given.

Another method of expressing corrosion test results which is particularly useful in the case of some of the high-strength light alloys used for aircraft is the loss-of-strength method. To the aircraft engineer, results expressed in this manner are far more useful than would be loss-of-weight or penetration data. In fact, the loss of weight is usually negligible and the penetration can be obtained only by a laborious metallographic study of polished cross-sections. Although Committee B-3 has not included this method in its recommendation, it is using it in the outdoor exposure tests which the committee is conducting.

It appears to the authors, therefore, that a single method to be followed rigorously in expressing corrosion data is hardly advisable.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 252 (1931).

CORROSION TESTING METHODS

By H. E. Searle[1] and F. L. LaQue[2]

Synopsis

The authors draw attention to the practical advantages of field corrosion tests as an aid in selecting materials to be used in equipment that must resist corrosion. A simple and inexpensive device is described that has broad application in field corrosion testing. Based on eight years of experience in the use of this and other devices, it is considered to be the most practical yet developed. Examples are given to illustrate the accuracy with which performance in service has been predicted through its use. The suggestion is ventured that standardization of apparatus for field corrosion testing will be of greater immediate utility than a similar standardization of laboratory testing equipment.

The outstanding success that has attended the endeavors of this Society in developing standardized methods for determining the mechanical properties of metals has not yet been achieved with respect to the corrosion-resisting properties. The extensive programs of Committees A-5 on Corrosion of Iron and Steel, A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, B-3 on Corrosion of Non-Ferrous Metals and Alloys, and D-14 on Screen Wire Cloth have not yet yielded a simple corrosion testing method that can be adopted as a standard by metal producers and consumers. There can be no doubt that this problem is difficult. None appreciate it better than the authors, who, in presenting this paper, have in mind that a résumé of their experiences, in the field of corrosion testing, perhaps may be helpful in speeding the development of standardized corrosion testing apparatus.

The primary object of a standardized corrosion test is to provide a means for determining the performance of materials in service. It is pertinent to note that the field corrosion tests carried out by Committees A-5, A-10, B-3 and D-14 probably have been of more practical value than the other activities of this Society in connection with corrosion testing.

Viewing the subject broadly, there are several possible testing methods, each of which has certain advantages and disadvantages that demand consideration in selecting the most appropriate method.

One testing method is to construct either small scale or full scale equipment that can be operated experimentally under the same conditions as would exist in equipment that is to be built. Here the effects of corrosion may be observed directly. Unfortunately, the cost of this method in money and time is likely to be high, especially when a number of metals are to be studied.

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[1] Manager, Sales Research, The International Nickel Co., Inc., New York City.

[2] Member, Development and Research Dept., The International Nickel Co., Inc., New York City.

What might be considered a modification of this method is the trial installation in existing equipment of some part or parts made from the metals to be studied. This has the disadvantage of introducing possible galvanic effects, which are apt to make interpretation of test results difficult and uncertain. This difficulty is not insurmountable, but its elimination is, to say the least, often inconvenient if not impractical.

There remains the common practice of exposing relatively small specimens to corrosion and noting results, from which conclusions may be drawn. The testing may be done either in the laboratory or in the field.

As first consideration should be given to whether the testing can be carried out to best advantage in the laboratory or in the field, it should be useful to consider the advantages and disadvantages of the two classes of tests.

COMPARISON OF FIELD AND LABORATORY TESTS

The principal advantage of the laboratory test is that the conditions of the test may be controlled. The great importance of aeration, temperature, velocity, etc., has been discussed by numerous investigators.[1] Several types of laboratory corrosion tests have been developed that make provision for controlling the conditions. An interesting example, which incorporates the experience of many years of corrosion study, is shown in Fig. 1. This device has been described by Fraser, Ackerman and Sands[1] and its predecessor by Thompson and McKay.[2] With this apparatus, which is applicable to the study of corrosion by liquids, one may study the effect of temperature or some other factor on the corrosion rate of samples of some standardized metal, or one may set up a standardized set of conditions and note the effect of varying the composition, finish, etc., of the metal.

The fact that the various factors affecting corrosion may be subjected to control in the laboratory test makes this test of inestimable value in fundamental studies of corrosion. Also, since one may set up a standardized set of conditions against which metals may be tested, the laboratory test is of very great value in research work leading to the development of new and improved materials. The salt-spray test[1] and the Strauss test[2] are useful in such research and in the inspection of metals, alloys, and protective coatings as well.

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[1] W. S. Calcott, J. C. Whetzel and H. F. Whittaker, "Corrosion Tests and Materials of Construction for Chemical Engineering Apparatus," *Transactions*, Am. Inst. Chemical Engrs., Vol. 15, Part I, pp. 3–169 (1923).

W. G. Whitman and R. P. Russell, "The Acid Corrosion of Metals—Effect of Oxygen and Velocity," *Industrial and Engineering Chemistry*, Vol. 17, pp. 348–354 (1925).

F. N. Speller, "A Study of Corrosion Factors and the Electrochemical Theory," *Industrial and Engineering Chemistry*, Vol. 17, pp. 339–346 (1925).

U. R. Evans, "The Fundamental Principles of Corrosion," *Chemistry and Industry*, Vol. 45, pp. 504–508 (1926).

O. B. J. Fraser, D. E. Ackerman and J. W. Sands, "Controllable Variables in the Quantitative Study of the Submerged Corrosion of Metals," *Industrial and Engineering Chemistry*, Vol. 19, pp. 332–338 (1927).

R. J. McKay, O. B. J. Fraser and H. E. Searle, "Practical Application of Corrosion Tests—Resistance of Nickel and Monel Metal to Corrosion by Milk," *Technical Publication No. 192*, Am. Inst. Mining and Metallurgical Engrs. (1929).

[2] J. F. Thompson and R. J. McKay, "Control of Motion and Aeration in Corrosion Tests," *Industrial and Engineering Chemistry*, Vol. 15, pp. 1114–1118 (1923).

The principal disadvantage of the laboratory test is a very practical one. It is the difficulty of discovering not only just what are the conditions as to solution composition, aeration, agitation, etc., under which the material is to be used in practice, but also of reproducing the conditions accurately in the test. An example is the case of sewage-handling equipment where the chemical composition of the sewage is certainly indefinite and is subject to all sorts of changes. If the conditions of the laboratory test do not duplicate the practical conditions accurately, then the interpretation of results in terms of the practical conditions becomes at once hazardous and difficult. In this connection there has been, unfortunately, too common a tendency to use a test procedure developed primarily for inspection purposes, such as the salt-spray test, for estimating the performance of materials under other and often unrelated conditions.

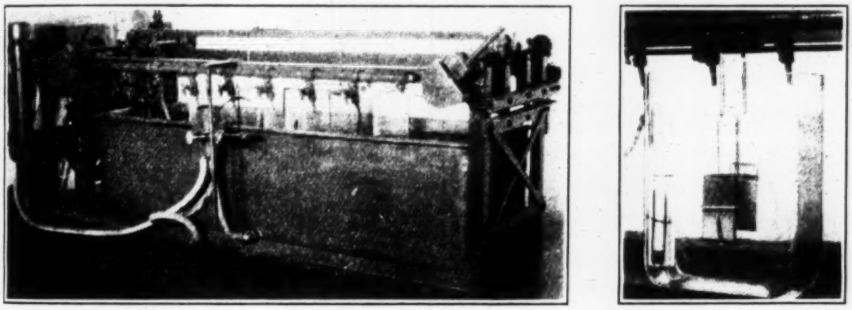

FIG. 1.—Circular Path Apparatus.

The complete apparatus is shown at the left. The method for supporting specimens and for aerating the test solution is shown at the right.

Another disadvantage is that, when large numbers of materials are to be tested, the time and equipment required are considerable and, consequently, the expense is great.

There are, in addition, some purely technical difficulties, such as the effect of corrosion products on the corrosive nature of the solution, and the possibility, especially with dilute solutions, of exhausting the corrosive constituents in the limited quantity of liquid that may be available.

The principal advantage of testing small specimens in the field is that the specimens are exposed automatically under the actual conditions of service, as the tests are made in equipment that is in service. This at once makes it unnecessary to discover just what the conditions are and it avoids the practical difficulty of reproducing them.

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[1] Protective Metallic Coatings for the Rustproofing of Iron and Steel, National Bureau of Standards *Circular No. 80*, pp. 24–30 (1919).

Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, pp. 209–214 (1932).

[2] E. C. Bain, R. H. Aborn and J. J. B. Rutherford, "The Nature and Prevention of Intergranular Corrosion in Austenitic Stainless Steels," *Transactions*, Am. Soc. Steel Treating, Vol. 21, pp. 481–509 (1933).

Another important advantage of field tests is that a large number of materials may be tested at the same time and under identical conditions. This facilitates direct comparisons, which are necessary in selecting materials. Furthermore, the material being used may be included in the test, which not only enables direct comparison with it but also comparison of the test result on that metal with its observed behavior. Thereby, a check is afforded against the test itself. The feasibility of testing a large number of specimens at one time results from a purely technical advantage of field tests, namely, the mass of corroding medium is usually very great in relation to that of the specimens, and, very often, the corroding medium is renewed either continuously or frequently, thus avoiding any important depletion of corrosives or change in their nature as a result of corrosion of specimens.

Furthermore, the test may be continued for as long as may be desired, with a minimum of expense. Little expert attention is required; in fact, field tests need not require the attention of anyone who is experienced in corrosion work.

While the practical advantages of field tests are very great, they are unsuited to certain purposes. For example, in fundamental studies of the effects on corrosion of variations in temperature, velocity, etc., the field test is not suitable, since the conditions of test are not subject to control. Moreover, it is necessary in some instances to determine the effect of corrosion on the corroding medium itself, as, for example, in the storage of distilled liquors. Obviously, a laboratory test is better suited to this sort of investigation than a field test, as the proper relationship between solution volume and exposed metal area may be reproduced easily in a laboratory test that nevertheless must duplicate practical conditions. In such cases an alternative procedure is to determine the corrosion rates by field tests, calculate the probable metal pick-up, and then make an independent laboratory investigation to determine the effects of varying amounts of the different metals on the liquid.

Occasionally the results of field tests are rendered uncertain with respect to some of the metals because corrosion products are introduced into the corrosive from the equipment in which the tests are made, in a quantity sufficient to influence the corrosion rate of certain metals being tested. For example, the corrosion rate of aluminum in an acetic acid still made of copper may be influenced by the presence of copper salts introduced as a result of corrosion of the copper. In such cases, supplementary information usually must be developed by laboratory methods.

Sometimes field tests disclose anomalies that cannot be investigated satisfactorily by further field testing. In a particular case it was found that milk being heated had a different corrosive effect from milk being cooled, although the other conditions of velocity of flow, temperature and aeration were identical. Laboratory tests soon showed that film formation on the metal surface was the controlling factor and they defined the conditions under which the film would form.

The foregoing examples illustrate the important point that field and laboratory tests may be used together to very good advantage in the study of corrosion. The field test is usually to be preferred when selecting a metal for some specific purpose, whereas the laboratory test is much better for studies of more fundamental nature. The two types of test are complementary and not competitive. Very often both types of test are necessary to a complete understanding of a corrosion problem.

FIELD TESTING METHODS

Recognizing that field tests have several substantial advantages and that they are more useful in practical work, the method for carrying them out becomes important.

The custom of making field tests by exposing specimens in operating equipment has been practiced for a long time. In its simplest form, it consists of hanging a piece of metal in a tank or other apparatus that is in operation. While this may yield useful results the hazards are great, as the specimens are likely either to be lost or to suffer mechanical damage. Furthermore, sufficient care may not be taken to avoid possible galvanic effects such as would result from the use of supporting wires of some other metal or from actual contact with other metals in the equipment in which the tests are made.

It seemed to the authors that it should be possible to develop some simple device that would avoid the hazards mentioned above and would, in addition, permit a comparison not only among different metals but also between metal couples and the same metals when not coupled. It was considered desirable to support the specimens so that the only way they could disappear from the holder would be by corrosion. For the sake of convenience it was decided that the holder should be made of standard materials that could be obtained readily.

At the same time, it was thought desirable to standardize the test further by using specimens of standard shape and size that could be made easily and mounted in such a way that a minimum percentage of specimen area would be in contact with the holder. Standardization of specimens has two important advantages: it permits the preparation in advance of many specimens of different metals that can be kept in stock so as to reduce the time required to get a test started, and it eliminates the necessity of measuring the area of each specimen, thereby eliminating much labor in calculating the corrosion rate in terms of weight loss, area, and time.

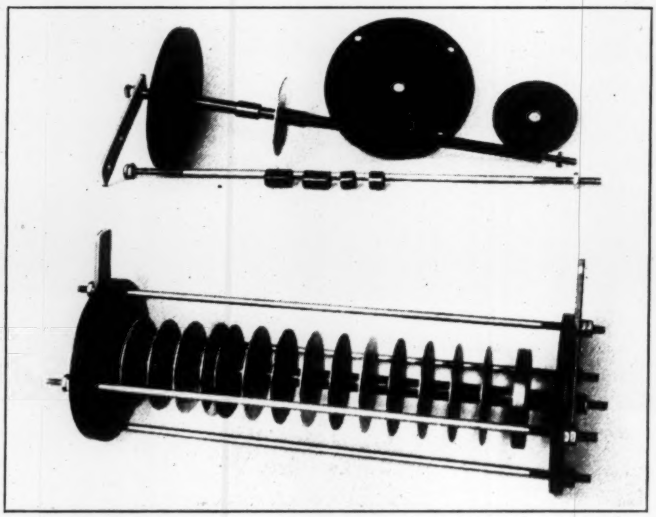

FIG. 2.—Spool-Type Specimen Holder.

BILL OF MATERIALS FOR ONE HOLDER

Number Required	Name of Part	Material and Size
5	Rods	Monel metal, $\frac{3}{16}$ in. in diameter by 12 in., thread ends $1\frac{1}{4}$ in., use 10/32 threads.
20	Hex Nuts	Monel metal machine screw nuts, No. 10/32.
2	Brackets	Monel metal, 11 U. S. Standard gage, $\frac{3}{4}$ in. by $3\frac{3}{4}$ in., drill 3 holes[a] $\frac{7}{32}$ in. in diameter.
1	Insulator	Molded bakelite tubing $\frac{5}{16}$ in. in outside diameter by $\frac{13}{64}$ in. in inside diameter by $10\frac{3}{4}$ in.
2	End Disks	Molded bakelite sheet 4 in. in diameter by $\frac{1}{4}$ in., drill 5 holes.[b]
15	Spacers	Molded bakelite tubing, $\frac{7}{16}$ in. in outside diameter by $\frac{21}{64}$ in. in inside diameter by $\frac{5}{8}$ in.
1	Short spacer	Molded bakelite tubing, $\frac{7}{16}$ in. in outside diameter by $\frac{21}{64}$ in. in inside diameter by $\frac{5}{16}$ in.

[a] Locate center of end hole $\frac{1}{2}$ in. from end on long axis, other hole centers are respectively $1\frac{3}{4}$ in. and $2\frac{3}{4}$ in. from center of first hole.

[b] Drill hole $\frac{21}{64}$ in. in diameter at disk center and locate centers of other holes, $\frac{7}{32}$ in. in diameter, at four points of quadrant and $1\frac{3}{4}$ in. from disk center.

AREAS OF STANDARD SPECIMENS ACCORDING TO THICKNESS

U. S. Standard Gage	Thickness, in.	Area Exposed		U. S. Standard Gage	Thickness, in.	Area Exposed	
		sq. dm.	sq. in.			sq. dm.	sq. in.
3	0.2500	0.599	9.286	20	0.0375	0.503	7.795
7	0.1875	0.571	8.847	21	0.0344	0.502	7.773
11	0.1250	0.543	8.409	22	0.0313	0.500	7.752
12	0.1094	0.535	8.299	23	0.0281	0.499	7.729
14	0.0781	0.521	8.080	24	0.0250	0.497	7.707
16	0.0625	0.514	7.970	25	0.0219	0.496	7.686
18	0.0500	0.509	7.883	26	0.0188	0.494	7.664

The Spool-Type Specimen Holder

The type of specimen holder that was arrived at finally, and which incorporated all the desired features, is illustrated in Fig. 2, with which a bill of materials is shown. For want of a more appropriate name, the device has been called a spool-type specimen holder. The specimens are machined to an exact diameter of 2.233 in. with a $\frac{21}{64}$-in. diameter hole in the center, and the standard thickness is $\frac{1}{32}$ in. This specimen, when mounted on the holder, has an exposed area of 0.500 sq. dm. Sometimes it is more convenient to use thicker specimens in which case the thickness may be measured and the corresponding area taken from the table shown with Fig. 2. The specimens can be weighed readily on a chemical balance, and, because of their uniform size, the effort involved in changing weights on the balance pan is minimized. This is important when weighing large numbers of specimens.

For certain purposes special specimens may be prepared, as, for instance, specimens made by welding together pieces of metal. Also special specimens containing soldered joints, etc., may be prepared and mounted on the spool-type specimen holder.

In assembling specimens for test, a supporting bracket is slid over the threaded end of the center rod and two nuts are put on and locked together so as to avoid loosening. Then an insulating tube is slid over the rod and next a large end disk which bears against the bracket. Next, an insulating spacer is slid over the insulating tube, then a specimen, then another spacer, another specimen, and so on until all the available space has been utilized. It is customary to mount at least two specimens of each metal. Then the second end disk is put on, followed by the other supporting bracket and nuts which are drawn up tight and locked. Four additional rods are inserted in the holes located around the peripheries of the end disks and these are drawn up tightly with nuts, which are locked. These four rods serve two purposes: they afford protection for the specimens against mechanical abuse, and they reinforce the assembly making it very strong. One of these rods passes through holes in the supporting brackets, thus serving to prevent motion of the brackets relative to the end disks, which might tend to loosen the nuts used to hold the apparatus together.

In order to provide for any variations in thickness of specimens, short spacers are available so that it is always possible to have the end disks project sufficiently beyond the sheathing tube to enable the assembly to be tightened properly.

When it is desired to investigate galvanic effects, the insulating spacer may be replaced by a metal spacer of the same or shorter length made from one of the metals in the couple. Ordinarily the metal spacer is considered part of the metal specimen comprising one half of the couple, but, if it be considered desirable, the exposed surfaces of the spacer may be covered with a coating of insulating lacquer. Similarly, corrosion of the specimens forming the couple may be confined to adjacent faces by lacquering the backs. When a galvanic arrangement of this kind is used, the practice is to use a compression spring of corrosion-resisting metal placed between a spacer and the end disk. This provides a means for taking up automatically any slack that possibly may result from thinning of specimens by corrosion. This assures continuous metal to metal contact. Of course this method of making galvanic tests is confined to one particular ratio of electrode areas and consequently the results are directly applicable only to practical exposures where a similar area ratio exists. Whitman[1] and others have discussed the importance of the electrode-area relationship.

Originally, it was hoped that the spacers would be made accurately enough to enable the assembly to be tightened so that no seepage would occur between specimens and spacers. However, experience has shown that seepage must be expected. This has proven to be more useful than not, since an opportunity is provided for concentration cells[2] of either the metal ion or oxygen type to become established at the spacers, thus affording a means for observing the susceptibilities of the metals tested to the localized corrosion produced by either type of cell. An idea of the susceptibilities of metals to concentration cell attack may be of as great importance as a knowledge of its over-all resistance to corrosion. At the same time, the percentage area of the specimen that takes part in these side reactions is relatively so small (about 1 per cent) that no important error is introduced in the measurement of corrosion on the freely exposed portions of the specimens.

The standard spacing of specimens is $\frac{5}{8}$ in., which is determined by the length of the spacer. The question has been raised occasionally as to the possibility of galvanic effects between adjacent specimens when the holder is submerged in liquids that are conductors, in spite of the insulating spacer. Those raising this question evidently overlook the impossibility of equal currents flowing through an electrolyte in two opposite directions at the same time from two electrodes that take part in the chemical reaction producing the current. Aside from these theoretical considerations experience has shown conclusively that no such galvanic effects occur. For example, specimens of aluminum and copper have been exposed adjacent to each other on the same spool under conditions where copper has corroded and aluminum has not, yet, when the insulating spacer was replaced with a copper spacer, the aluminum corroded more than the copper under the influence of the galvanic current.

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[1] W. G. Whitman and R. P. Russell, "The Natural Water Corrosion of Steel in Contact with Copper," *Industrial and Engineering Chemistry*, Vol. 16, pp. 276–279 (1924).

W. G. Whitman and R. P. Russell, "Fundamental Factors of Corrosion," *Chemical and Metallurgical Engineering*, Vol. 30, pp. 671–672 (1924).

[2] R. J. McKay, "Corrosion by Electrolyte Concentration Cells," *Transactions*, Am. Electrochemical Soc., Vol. 41, p. 201 (1922).

R. J. McKay, "The Common Occurrence of Corrosion by Electrolyte Concentration Cells," *Industrial and Engineering Chemistry*, Vol. 17, pp. 23–24 (1925).

Of course the choice of materials from which to make the various parts of the holder is important. While they do not need to be durable enough to last indefinitely, they must last long enough to insure satisfactory completion of the test. The metal parts have been made generally of nickel or monel metal for exposure to the majority of corrosives and of 18 per cent chromium, 8 per cent nickel stainless steel for tests in oxidizing acids, such as nitric. The insulating materials used have been bakelite, porcelain and glass, with bakelite answering most purposes. Its principal limitations have been temperatures over 300 F., concentrated alkalies and certain organic compounds such as coal-tar products.

Experience has shown that it costs about 20 cents to machine a specimen and roughly $3 to buy material and make the parts of the spool-type holder, when about 25 holders are made at one time.

Specimens are prepared for test by resurfacing if desired, scrubbing, rinsing in water and alcohol-ether mixture, and drying. Weighing is done with an accuracy of ± 0.0001 g. After test, any adhering corrosion products are removed, usually by brushing or rubbing. Then the specimens are rinsed, dried and weighed as before. The closeness of checks between weight loss of different specimens of the same metal on the same spool is very good. Variations from the average of more than 5 per cent are rare and much closer checks usually are obtained.

Although loss in weight is used as the principal measure of corrosion, this is supplemented always by an examination of each specimen for tarnish, nature of corrosion product, etc., before it is cleaned. If pitting has occurred, the maximum, minimum and average pit depth is determined by means of a calibrated microscope, which is focused on the edges and then the bottoms of the pits in the weighed specimen. Distinction is made between pits in the surfaces that are exposed freely and the surfaces under the spacers. Then the specimen may be subjected to bending tests and microscopic examination to determine whether intergranular corrosion or dezincification has occurred. The standard specimens are not well adapted to the quantitative measurement of change in mechanical properties.

The method of supporting the specimen holder during test is important. The preferred position of the holder is with the long axis horizontal, which avoids dripping from one specimen to another. The holder must be located so as to cover the conditions of exposure that are to be studied. It may need to be submerged, or exposed only to the vapors, or located at the liquid level. The last may be accomplished most readily, when the liquid level is changing, by building a suitable float, usually of wood, around the holder so that it will float on the surface. Various means have been utilized for supporting the holder in liquid or vapor. The simplest is to suspend the holder by means of heavy wire or light metal chain. Holders have been strung between heating coils, clamped to agitator shafts, bolted to evaporator-tube sheets, etc.

The spool-type specimen holder was developed by the authors about eight years ago, and since that time it has been used by them, their associates and others in testing at least 10,000 specimens under a wide variety of conditions. At this writing, there are in test some 1500 specimens in 100 locations.

ACCURACY IN PREDICTING PERFORMANCE

The accuracy with which tests made in this manner have predicted performance in service will be of interest, since this is the true measure of the usefulness of the testing method and the device. A few typical examples taken from many will serve to indicate what has been found generally to be true.

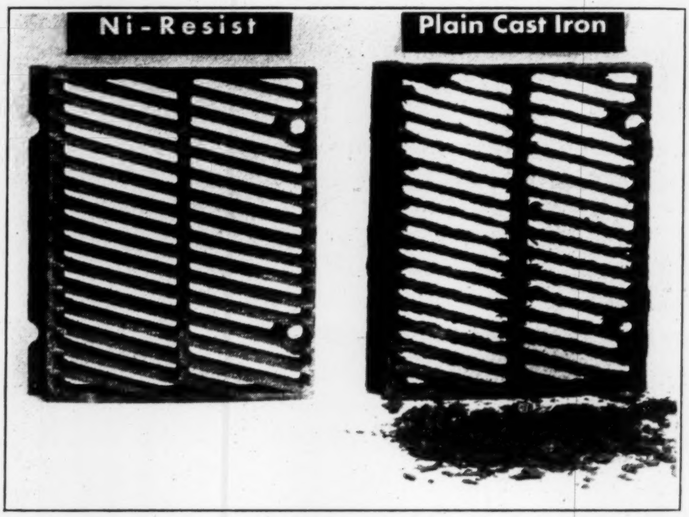

FIG. 3.—Comparison of Ni-Resist Grid After 18 Months of Service and a Plain Iron Grid After 4 Months of Service.

A salt company was experiencing considerable trouble with corrosion of cast iron filter support grids on a rotary filter-drier used for de-watering and drying salt in one operation. The corrosion was aggravated and complicated by suspected galvanic effects between the metallic filter cloth and the grids. As the cycle of operations involved exposure to saturated brine that was first cold and then hot and, finally, exposure to a blast of hot air, it was obviously difficult to reproduce conditions in the laboratory but quite convenient to place a spool immediately beneath a grid in the operating machine. The test was allowed to run for 5 months during which it required no attention. The results obtained indicated that the alloy cast iron Ni-Resist would corrode at only one-twelfth the rate of plain cast iron. Also, the results showed that coupling these irons with the metal of the filter cloth approximately doubled corrosion in each case. From this it appeared that the Ni-Resist should outlast the plain iron 12 times, or for 48 months as compared to the 4 months being obtained from plain cast iron. Figure 3 shows the condition of a Ni-Resist grid removed for inspection after 18 months of service and beside it is a plain iron grid removed after 4 months of service. By April 1935, the Ni-Resist grid has outlasted the plain iron grid over 9 times and it is evident from inspection that the predicted service is likely to be attained.

In another case, tests made with the spool-type holder indicated that the metal pick-up of a fruit juice from the metal storage equipment, in which the tests were made, would be 49 p.p.m. after a certain period. Actual analysis by a consulting chemist of juice stored for this period showed 53 p.p.m., which checked closely with the estimated pick-up. Similar calculations have been made in the case of copper equipment used for making condensed milk.[1] The calculated copper content was found to lie within the range reported by another investigator who analyzed a variety of typical samples of condensed milk.

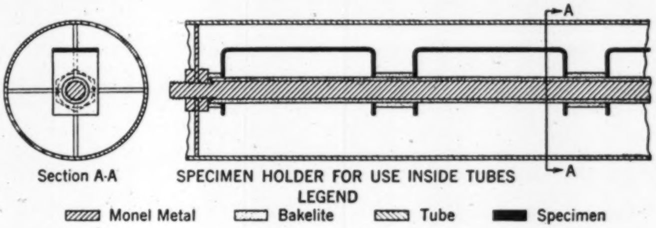

Fig. 4.—Special Field Testing Device for Testing Specimens Within Tubes.

Special Specimen Holders

While the standard spool-type specimen holder serves very well in most corrosion tests that are carried out in the field, there are instances where special holders are necessary. For example, it is impossible to put the spool-type holder in a small pipe line or to use it in comparing materials for tie rods of wooden tanks, which are usually buried in the wood.

As corrosion on the inside of pipe lines is often important, several types of specimen holders were tried out in this type of work. Experience soon demonstrated that most of them had important disadvantages. The most appropriate arrangement turned out to be a variation of the spool-type holder in which the shape of the specimen was changed from a disk to a strip $\frac{1}{2}$ in. wide by 3 in. long, bent at each end, as indicated in Fig. 4 and provided with $\frac{21}{64}$-in. diameter holes in the bent portions. Specimens of this kind may be assembled and spaced on a rod of the same diameter as used in the standard holder and in the same manner. By replacing the end disks with spiders made by twisting wire, the specimens may be located in the center of the tube. This device does not interfere seriously with the flow of liquid through tubes $1\frac{1}{2}$ in. in inside diameter or over. By means of this device, tests have been made to compare corrosion rates of metals in sanitary pipe lines handling milk, and in brine lines.

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[1] E. C. Thompson, R. P. Mears, H. E. Searle and F. L. LaQue, "Metals and Condensed Milk—Corrosion of Metals in Manufacture of Evaporated and Sweetened Condensed Milk," *Industrial and Engineering Chemistry*, Vol. 25, pp. 1311–1316 (1933).

Other devices have had to be developed for use in more specialized cases. For example, it was desired to compare the performance of metals that might be used to line rotary driers commonly employed for drying wet salt crystals. In this case, the investigation involved not only corrosion but abrasion as well, and the device used to hold the specimens had to be such that the specimens would be subjected to both in the same manner as a drier lining. By using the standard specimens of the spool-type holder and bolting them flat to a board of pressed asbestos substance and then bolting the whole arrangement to conform with the inside surface of the drier, results were obtained that gave a reliable comparison of the different metals. The tests indicated a maximum rate of penetration of 0.012 in. per yr. for the metal being used in the lining. By actual measurement of lining thickness in 3 driers, the maximum yearly penetration was found to be 0.0118 to 0.0127 in. With such a check against known performance, the results indicated by the test for other metals are likely to be reliable.

CONCLUSIONS

In conclusion, the authors hope that this outline of the desirability of field tests and the description of testing methods and devices that have been found useful will increase interest not only in field tests but in the standardization of field-testing apparatus. Based on eight years of experience with the devices that have been described and with others as well, the spool-type specimen holder is the most practical device yet developed for use in field corrosion testing. The spool-type specimen holder is simple and inexpensive. It is applicable to a broad range of field testing, including the exposure of different metals, either plain or welded and either singly or in galvanic combination, to wet solids, liquids and vapors. Test data are obtained at low cost and with a minimum of expert attention. The performance of materials in corrosion services is predicted accurately. The authors venture the suggestion to the Society that a standardization of apparatus for field corrosion testing will be of greater immediate utility than a similar standardization of laboratory testing equipment.

THE CONTROL OF CORROSION IN AIR-CONDITIONING EQUIPMENT BY CHEMICAL METHODS

By C. M. Sterne[1]

Synopsis

With the increasing use of air conditioning, it is timely that serious thought be given to one of the most vexing problems that appears only after the equipment is in operation for some time, namely, corrosion.

Corrosion attack of the metallic parts of air-conditioning systems can be so severe that perforation of ferrous sheets and pipes may take place in as short a time as four months if the wash water or condenser water is not chemically treated. Practically every type of corrosion attack is common to these systems. Service corrosion tests and analyses of the recirculated water show very clearly the causes and degree of severity of that attack. The tests also show the degree of protection that may be obtained by proper chemical treatment. Corrosion tests have been run by the author in over 200 separate air-conditioning systems, handling both comfort and industrial conditioned spaces in many types of industry and in various cities throughout the United States. Representative cases are reported in this paper.

Check examinations of these systems by the author and others have shown conclusively that by proper chemical treatment and supervision the corrosion attack may be reduced to a minimum.

Introduction

Air conditioning is coming into such common use that the corrosion problems encountered in connection with the operation of this type of equipment are becoming of utmost importance. The various types and forms of corrosion found in the recirculated water phases of air-conditioning equipment and the severity of attack present a general problem to both the manufacturers and the owners of this type of equipment, as well as to the metallurgist, chemist and corrosion engineer who is consulted, if the equipment is to be preserved. The permanence of this equipment is of great concern to the owner, who for the most part is frequently not aware of the existence of a corrosion problem and who is generally not familiar with methods used to control this corrosion. This has resulted in numerous costly failures in a very short time. The large manufacturers of air-conditioning equipment and some consulting engineers and architects have recognized this problem and are taking steps to see that their customers and clients are acquainted with these facts and are advising chemical treatment of the recirculated water.

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[1] Chief Engineer, Metropolitan Refining Co., Inc., Long Island City, N. Y.

AIR-CONDITIONING EQUIPMENT

To better understand the physical make up of an average air-conditioning system, a schematic diagram (Fig. 1) and short explanation of the cycles of the recirculated wash water and condenser-cooling tower circuit are given.

During the refrigeration season (hot weather), the wash water is sprayed in the dehumidifier, returns to the cooler and is then pumped back to the dehumidifier to be resprayed. In the dehumidifier, *A*, water is sprayed from opposing banks of nozzles, whose sprays impinge upon each other. In the winter season the recirculation is only through the dehumidifier and circulating pump, the dehumidifier acting as a humidifier. In large air-conditioning systems any number of dehumidifiers and refrigerating machines may be connected together, depending upon the specific requirements. The temperature of the wash water ranges from 34 F. to 70 F. to meet varying operating and seasonal conditions.

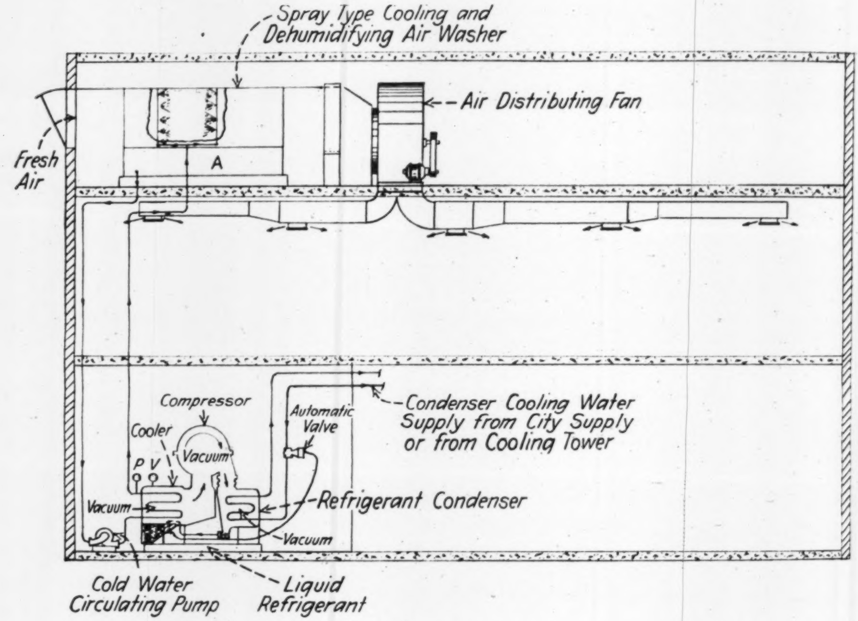

Spray Type Cooling and Dehumidifying Air Washer
Air Distributing Fan
Fresh Air
A
Compressor
Cooler
Vacuum
P V
Automatic Valve
Condenser Cooling Water Supply from City Supply or from Cooling Tower
Vacuum
Refrigerant Condenser
Vacuum
Cold Water Circulating Pump
Liquid Refrigerant

FIG. 1.—Schematic Layout of a Central Station Air-Conditioning System.

The condenser water goes from the condenser to the cooling tower, where it is sprayed and then flows back to be pumped through the condenser. The water temperatures range from 60 F. to 150 F.

THE CORROSION PROBLEM

It will readily be seen that in a central station air-conditioning system the corrosion exposures are of the following types:

1. Spray (*a*) alternate, (*b*) continuous;
2. Humid atmosphere (*a*) alternate, (*b*) continuous;
3. Water line (*a*) agitated, (*b*) quiescent;
4. Velocity;
5. Impingement;
6. Acid (due to air contamination);
7. Galvanic action (due to different metals in contact); and
8. Electrolytic action (in rare instances, due to current leakage).

The chief characteristics of the recirculated water affecting the rate of corrosion are:

1. The softness of the water,
2. The chloride content,
3. The carbon dioxide content,
4. The temperature of the water,
5. The oxygen content,
6. The sulfate content, and
7. The pH value of the water.

In the problem under discussion, the more important factors are the temperatures, oxygen concentration, sulfate content and pH value. With regard to oxygen content, the recirculated wash water is completely saturated at all times after coming from the air washer, and with the exception of vacuum water vapor refrigeration systems, practically none of this oxygen is removed. It has not been found practicable to install deoxygenating or deactivating equipment or to employ chemicals such as sodium sulfite for oxygen removal. The washing of impure air usually results in a large increase in the sulfate content and a drop in the pH value. The analyses given in Table I illustrate this statement. It will be noted that in the New York City water the pH value dropped from 7.0 to 3.3, showing the development of an acidity 7000 times as great as that initially present. At the same time the sulfate content increased approximately 79 times. The increase in iron content of the water is indicative of corrosion taking place due to this contamination.

A further proof that the quality of the air is probably the most important factor contributing to excessive corrosion is shown by experience with water from a large central station air-conditioning system in Chicago. In the fourth column of Table I is given the analysis of raw water used in the ten dehumidifiers of the system. The outside air supply is taken from two separate sources in different sections of the building. Five of the dehumidifiers are connected to each fresh-air intake. The analysis in the fifth column of Table I is that of the water from one of the five dehumidifiers getting contaminated air where the corrosion attack has been so severe that the casing walls were perforated in less than 4 months. The other five dehumidifiers receiving uncontaminated air are in good condition. The analysis of this water is shown in the last column of Table I. During the refrigeration season the entire system, however, will be subject to an accelerated corrosion attack due to mixing of all the water in the system, because all of the water comes back to a common surge tank before going through the refrigerant coolers. It is of course not generally possible to change the local atmosphere, therefore it is necessary to use other methods to protect successfully the parts of the equipment in contact with this water.

MATERIALS OF CONSTRUCTION

The dehumidifier tank is usually made of No. 16-gage bare copper-bearing steel, the casings of No. 18-gage galvanized copper-bearing steel (with all seams and rivets soldered) and the baffle and eliminator plates of No. 24-gage galvanized copper-bearing steel. The eliminator and baffle plates extend below the water level for about 3 in. and are exposed to corrosion attack on both sides. Wrought or cast iron is used for water delivery headers, bare or galvanized steel pipe for spray risers, galvanized steel for spray-head nipples and bronze for spray heads. Bare or galvanized-steel pipes connect the dehumidifiers to the coolers or evaporators. The pumps have cast iron casings and bronze or iron pump impellors. The coolers and condensers usually have steel or cast iron shells and water boxes and steel, wrought iron, admiralty or Muntz metal tubes and tube sheets.

TABLE I.—CHANGES TAKING PLACE IN WATER DUE TO RECIRCULATION WITH VARIOUS ATMOSPHERES.

	Average New York City Raw Supply to System	New York City, in System 6 Days	Average Chicago Water Raw Supply to System	Chicago, in System 48 hr.	
				Contaminated Air	Uncontaminated Air
Silica, p.p.m.	2.03	2.53	4.2	4.4	4.2
Iron and aluminum oxide, p.p.m.	0.43	475.33	2.2	115.0	3.2
Calcium oxide, p.p.m.	8.81	37.01	47.0	124.0	48.0
Magnesium oxide, p.p.m.	2.02	6.45	18.0	49.0	20.0
Bicarbonates as HCO_3, p.p.m.	7.51	......	102.0	23.0	95.0
Carbonates as CO_3, p.p.m.	....	......	8.5	....	11.0
Chlorides, p.p.m.	3.32	19.50	12.0	28.0	3.8
Sulfates, p.p.m.	6.03	476.18	17.0	355.0	29.0
pH value	7.0	3.3	7.7	4.7	7.7

Readily accessible portions of the equipment are usually painted—in most cases with asphaltum paints. While asphalt in itself is more or less inert to alkalies and acids, practically all of the asphalt paints are cut back with thinning oils as a vehicle which are readily attacked by strong alkalies and acids. Due to their porosity their life expectancy is relatively short, necessitating frequent repainting. The new resin and lacquer coatings, while still high in price, may prove applicable to this type of work, but little experience in their use is yet available. Hydrogenated oils may have some possibilities but so far have been little used. Unfortunately, it is not possible to paint the insides of small pipes nor is it desirable to put insulating paint coatings on heat transfer surfaces. Cement-lined pipes offer too many construction and erection difficulties, as do artificial stoneware and glass pipes. A pyrex glass dehumidifier would be too high-priced to command a ready sale. Glass or stoneware spray nozzles are used extensively in some industrial air-conditioning installations. Metallic coatings applied by the spraying process are in general too porous to offer good protection in air-conditioning equipment and the cost is excessive if sufficient thickness of coating metal is applied to give adequate protection. Experience has shown that zinc coatings corrode very quickly in dehumidifiers and lines carrying the recirculated water, irrespective of the zinc application method, when the usual contaminated air is encountered.

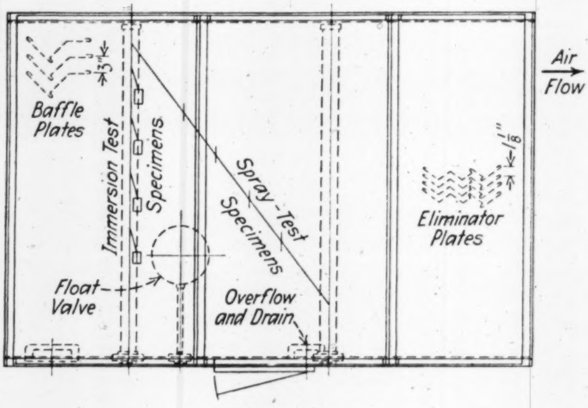
Electrolytic protection methods have not been successful in corrosion protection in air-conditioning equipment. This is due primarily to the size of the equipment and the difficulty of finding proper anode materials and so placing them as to distribute the current adequately.

In special jobs, practically every type of corrosion-resistant metal and alloy has been used at some time or other. Tanks, casings, etc., have in some cases been made of concrete, tile or wood. The prime cost of installations made of special corrosion-resistant alloys is too great to permit their extensive use and in some cases the selection of the wrong alloys has resulted in costly corrosion failures. In general, the ordinary ferrous alloys are used for this type of equipment, and are subject to rapid corrosion under the conditions encountered. Experience has shown that chemical treatment of the recirculated water offers the most practical method of controlling the corrosion. It is desirable to have a reliable rapid method of determining what type of chemical treatment will give the best corrosion protection.

METHOD OF TEST

In this paper a method of measuring the potential corrosion rate is presented and the effectiveness of various forms of chemical treatment in minimizing the corrosion occurring in ferrous metal air-conditioning systems is demonstrated by means of this method. Test specimens of low-carbon steel, copper-bearing steel, special alloys and stainless steels have been used. Also copper, 70-30 brass, admiralty metal, Muntz metal, monel and aluminum alloys have been tested at times, although this paper does not cover tests of the non-ferrous metals. Particular attention was paid to copper-bearing steel which is the metal most commonly used. The tests were made using a steel of the following analysis: carbon 0.073, manganese 0.42, silicon 0.044, phosphorus 0.018, sulfur 0.006, and copper 0.21 per cent.

The corrosion tests are carried out in the following manner: Individual test specimens 2 by 4 in. by 16 gage, punched at the top center for hanging, are first rough ground on a commercial endless belt polishing machine, using No. 80 Marathon cloth, care being taken that heating of the pieces does not take place during the grinding. They are then finished by hand using No. 00 emery cloth. The specimens are then degreased in an alcohol-ether mixture and air dried. They are then weighed on an analytical balance to a ±1-mg. accuracy. Four specimens are then hung by fish line in the tank water for a complete immersion test and four specimens for a spray test between the opposing banks of sprays. Care is taken to see that there is no contact between the individual test specimens or between the test specimens and any part of the equipment, as shown in Fig. 2.



Plan

FIG. 2.—Distribution of Test Pieces in Dehumidifier.

At the end of ten days the test specimens are removed from the equipment, cut from the fish line, towel dried and are then taken to the laboratory. They are degreased with carbon tetrachloride and cleaned using a 1:1 HCl solution containing 2 per cent No. 40 Rodine inhibitor, rinsed in water and dried, using an alcohol-ether mixture. They are then reweighed on the same balance used for the initial weighings. Blanks are run in the same inhibited acid mixture for the same time to detect any loss in weight due to cleaning. From the corrected weight loss and the area exposed the amount of corrosion is expressed in milligrams per square inch.

RESULTS

A large number of tests have been run during the past four years in air-conditioning equipment in buildings of banks, public utilities, research

TABLE II.—WEIGHT LOSSES IN MILLIGRAMS PER SQUARE INCH, AVERAGE IN EACH CLASS OF TEST.

Duration of tests 10 days. Average of 4 test specimens in each case.
Analyses of raw water and water at end of one week's run shown at each location.

	New York City 7 Days Use		Philadelphia 6 Days Use		Washington, D. C. 6 Days Use		Tobacco Plant[d] 7 Days Use		Rayon Plant 7 Days Use	
	Raw Water	Continuous	Raw Water	10 hr. (Day)	Raw Water	8 hr. (Day)	Raw Water	Continuous	Raw Water	Continuous
Silica, p.p.m.	2.1	2.5	4.8	5.1	7.4	4.5	13.0	27.0	3.6	18.0
Iron oxide, p.p.m.	0.4	475.3	5.1	86.8	1.8	26.2	1.8	63.6	1.1	374.0
Calcium oxide, p.p.m.	8.8	37.0	25.4	32.5	49.4	46.2	17	31.0	33.0	176.0
Magnesium oxide, p.p.m.	2.1	6.4	11.7	13.7	12.5	15.6	1.6	7.4	4.0	26.0
Bicarbonates, p.p.m.	7.5	.....	32.5	12.1	42.6	48.4	60.0	....	20.0	....
Carbonates, p.p.m.	....	.....	....	.....	37.8	.....	....	.....	.....	.....
Chlorides, p.p.m.	3.3	19.5	16.2	48.2	9.2	33.6	3.5	15.3	10.0	42.0
Sulfates, p.p.m.	6.1	476.2	24.8	116.3	11.2	54.7	21.0	82.0	25.0	670.0
pH value	7.0	3.3	7.1	4.9	7.5	6.1	7.2	4.7 (organic acids)	8.1	3.0
Treatment	Total Immersion	Sprays	Total Immersion	Sprays	Total Immersion	Sprays	Total Immersion	Sprays	Total Immersion	Sprays
None[a]	115.4	268.2	85.3	188.7	66.0	146.6	158.1	302.6	107.7	396.1
No. 1	0.4	0.5	0.4	0.4	0.4	0.4	0.5	0.6	0.8	0.9
No. 2	2.0	1.1	1.5	1.0	1.5	1.1	[c]	[c]	2.6	3.0
No. 3	8.2	7.5	7.4	7.0	[c]	[c]	16	22.1	18.3	25.6
No. 4[b]	54.6	70.1	46.1	59.3	[c]	[c]	59.	78.6	63.5	82.3
No. 5[b]	53.0	69.2	46.0	58.7	[c]	[c]	57	74.3	62.1	80.9
No. 6	37.4	32.4	[c]	[c]	[c]	[c]	[c]	[c]	[c]	[c]
No. 7	33.1	28.6	[c]	[c]	[c]	[c]	[c]	[c]	[c]	[c]
No. 8	16.1	17.3	[c]	[c]	[c]	[c]	[c]	[c]	[c]	[c]
No. 9	3.0	2.1	3.1	2.4	[c]	[c]	[c]	[c]	[c]	[c]
No. 10[a]	110.6	255.0	[c]	[c]	[c]	[c]	[c]	[c]	[c]	[c]
No. 11[a]	113.0	256.3	82.9	182.3	[c]	[c]	[c]	[c]	[c]	[c]
No. 12[b]	52.6	67.1	[c]	[c]	[c]	[c]	[c]	[c]	[c]	[c]
No. 13[b]	51.7	65.2	46.2	59.1	[c]	[c]	[c]	[c]	[c]	[c]
No. 14[b]	45.2	59.3	[c]	[c]	[c]	[c]	[c]	[c]	[c]	[c]

[a] General heavy corrosion on test pieces.
[b] Total immersion test specimens in all tests in all locations using treatments Nos. 4, 5, 12, 13, 14 badly pitted.
[c] No test run.
[d] 6 p.p.m. chlorine for algae and fungii control, with periodic dosages of 5 p.p.m. copper sulfate used in tobacco plant air-conditioning equipment.

NOTE.—The treatments referred to were as follows:

Treatment	Concentration of Main Constituents in Recirculated Water	Average pH Value in Recirculated Water During Test	Solution Used
No. 1	500 p.p.m. sodium dichromate	7.5 - 8.0	(1) 42 per cent sodium dichromate, 6 per cent sodium hydroxide. (2) 40 per cent sodium hydroxide, 9 per cent sodium dichromate
No. 2	350 p.p.m. sodium silicate as SiO_2 120 p.p.m. sodium dichromate	8.0 - 8.5	28.3 per cent sodium silicate, 4.5 per cent sodium hydroxide, 3.2 per cent sodium carbonate, 9.1 per cent sodium dichromate, 1 per cent trisodium phosphate
No. 3	50 p.p.m. sodium dichromate	8.0 - 9.0	40 per cent sodium hydroxide, 9 per cent sodium dichromate
No. 4		8.0 - 9.0	48.3 per cent sodium hydroxide
No. 5		8.0 - 9.0	20 per cent sodium carbonate
No. 6	40 p.p.m. sodium silicate as SiO_2	8.5 - 9.0	58° Baumé sodium silicate
No. 7	55 p.p.m. sodium silicate as SiO_2	8.5 - 9.0	52° Baumé sodium silicate
No. 8	100 p.p.m. sodium silicate as SiO_2	7.5 - 8.0	27.4 per cent sodium silicate, trace tannin
No. 9	500 p.p.m. sodium silicate as SiO_2	8.5 - 9.0	40° Baumé sodium silicate
No. 10		7.0 - 3.9	Tannin and other undetermined organic material—solution added at rate of 1 qt. to 35,000 gal. of water
No. 11		7.0 - 3.8	Tannin, iron, undetermined organic matter—solution added at the rate of 1 qt. to 35,000 gal. of water
No. 12		8.0 - 9.0	Treatment No. 11 and sufficient 50° Baumé sodium hydroxide to maintain average pH value
No. 13		8.0 - 9.0	Treatment No. 11 and sufficient sodium carbonate to maintain average pH value
No. 14	300 p.p.m. sodium phosphate as PO_4	7.5 - 8.5	Tri-sodium phosphate

NOTES:

The difference in weight losses between the four test specimens in each type of exposure in the individual tests were remarkably small. For instance, in the New York City test without treatment, the individual losses were: specimen 1, 115.9; specimen 2, 115.0; specimen 3, 115.2; specimen 4, 115.6; average 115.4 mg. per sq. in. The spray-test specimens without treatment showed slightly larger differences in weight loss between the individual test specimens. In tests showing very small average weight losses, the differences were, of course, minute.

Tests were run on various other available ferrous alloys; the results obtained under complete immersion conditions were substantially the same as those given above. In the spray test the copper-bearing alloys showed slightly less loss in tests without treatment, but under conditions of adequate chemical treatment the results were substantially the same for all ferrous alloys tested.

The additions of sodium hydroxide and sodium carbonate where other active constituents were used were for the purpose of maintaining the desired pH value in the recirculated waters.

All of these tests were carried out in large commercial air-conditioning systems, and no changes were made in the normal operating procedure during the period of the test.

organizations, universities, government agencies and in many industrial plants, representing a wide variety of water supplies and conditions of air contamination, using the above method of test. Some of the representative data are presented in Table II to illustrate the effectiveness of various chemical treatments in minimizing corrosion.

It will be observed from Table II that treatments Nos. 10 and 11 were ineffectual, as the weight losses were essentially the same as when no treatment was used.

Treatments Nos. 4, 5, 12, 13 and 14 somewhat reduced the weight loss, but the test specimens were badly pitted. These are essentially alkali treatments except in the case of No. 14, where the phosphates may be considered as having film-forming properties. If the alkalinity of the wash water is further increased, there is not only the danger of corrosion attack of zinc and the zinc-bearing non-ferrous alloys, but the water is so softened that foaming takes place in the dehumidifier and some of this foam is blown through the eliminators and is carried into the ducts. This is especially true of treatments consisting mainly of sodium hydroxide or carbonate.

As the concentration of sodium silicate is increased, see treatments Nos. 6, 7, 8 and 9, the protection rapidly increases to a point where with 500 p.p.m. the protection is quite satisfactory due to the film-forming characteristics of the silicates.[1,2,3] However, there have been a number of cases where the silicates in this concentration have gone out of solution, especially in small-diameter cooler tubes, with a consequent plugging of these tubes. The silicate also acts as a binder on the eliminator and baffle plates for dirt, organic matter, etc., and by so doing has not only impaired the efficiency of the eliminators for taking care of entrained water but has been partly responsible for unpleasant odors from the collected decaying organic materials.

From Table II it may readily be seen that sodium dichromate (treatment No. 1) used in sufficient amounts gives the best protection of any of the chemical treatments tested. Also from treatment No. 3 it will be seen that small additions of sodium dichromate to a sodium hydroxide treatment greatly increased the amount of protection over that afforded by sodium hydroxide alone. These results might be expected because of the action of the dichromate as a passifying agent.[4] The question has been raised about possible carryover of chromates into the conditioned spaces. In one installation where dichromate treatment was used, samples of air were taken directly at the delivery ports. One sample showed 0.00066 mg.

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[1] F. N. Speller, "Corrosion, Causes and Prevention," Second Edition, p. 348, McGraw-Hill Book Co., Inc., New York City (1935).

[2] W. G. Whitman, E. L. Chappel and J. K. Roberts, "Corrosion in the Refrigerating Industry," *Refrigerating Engineer*, Vol. 12, pp. 158–165 (1926).

[3] R. B. Mears, Private communication.

[4] T. P. Hoar and U. R. Evans, "Passivity of Metals VII, Specific Function of Chromates," *Journal*, Chemical Soc. (British), Part II, pp. 2476–2481 (1932).

TABLE III.—INSPECTION DATA ON VARIOUS SYSTEMS.

Location	Treatment	Time Operated Before Examined, months	Condition of System	Remarks
New York City	none	6	very bad	Failure of casings and eliminators, general heavy corrosion, replacement of pipes.
New York City	No. 1	12, 24, 36, 48	excellent	No indication of corrosion.
New York City	No. 2	12, 24, 36, 48	excellent	No indication of corrosion, very thin continuous film visible when dry.
New York City	No. 3	12, 24	excellent	No indication of corrosion.
New York City	No. 4	12, 36, 48	poor	Repainted yearly, deep pitting, replacement of parts due to corrosion.
New York City	No. 5	24	poor	Galvanizing gone, deep pitting, replacement of parts due to corrosion.
New York City	No. 6	6	fair	Some pitting, indications of corrosion.
New York City	No. 7	6	fair	Light pitting, indications of corrosion, visible film when dry.
New York City	No. 8	12	good	Very slight pitting, traces of corrosion, visible film when dry.
New York City	No. 9	14	excellent	No indication of corrosion, cooler tubes blocked, necessitating 3 days shutdown.
New York City	No. 10	3	fair	New system, treatment No. 1 adopted.
New York City	No. 11	$2\frac{1}{2}$	fair	New system, treatment No. 1 adopted.
New York City	No. 12	3	fair	Treatment No. 2 adopted at end of 3 months.
New York City	No. 13	4	fair	Treatment No. 1 adopted at end of 4 months.
New York City	No. 14	18, 30, 48	poor	Repainted twice, deep pitting, replacement of parts due to corrosion.
Philadelphia	No. 1	12, 36	excellent	No indication of corrosion.
Philadelphia	No. 2	24, 36, 48	excellent	No indication of corrosion, very thin continuous film visible when dry.
Philadelphia	No. 11	24	poor	Pitting, repainting necessary twice.
Washington	No. 1	12	excellent	No indication of corrosion.
Tobacco plant	No. 1	12, 24[b]	fair[a]	System had previously had numerous replacements, corrosion now seems to be controlled.
Tobacco plant	No. 3	12[b]	fair[a]	System had previously had numerous replacements, corrosion now seems to be controlled.
Tobacco plant	No. 4	[b]	fair[a]	Owners' Research Department considered treatment No. 4, ineffectual; treatment No. 1 used.
Tobacco plant	No. 5	[b]	fair[a]	Owners' Research Department considered treatment No. 5, ineffectual; treatment No. 1 used.
Rayon plant	No. 1	12, 24, 36	excellent	No indication of corrosion, treatment used from beginning in new system.
Rayon plant	No. 2	12	excellent	No indication of corrosion, very thin continuous film visible when dry (new system).
Rayon plant	No. 3	12, 24	excellent	No indication of corrosion.
Rayon plant	No. 4	[c]	excellent	Owners' Research Department considered treatment No. 4, ineffectual, treatment No. 1 used.
Rayon plant	No. 5	[c]	excellent	Owners' Research Department considered treatment No. 5, ineffectual, treatment No. 1 used.

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[a] Old systems not previously treated.

[b] Systems previously in use about 8 to 10 yr. without treatment; almost completely rebuilt.

[c] New systems continuously treated with treatment No. 1 except during the 10-day tests of treatments Nos. 4 and 5.

sodium dichromate per 100 cu. ft. of air; all other samples showed negative. The wash water contained, at the time of the test, 504 p.p.m. sodium dichromate and 420 p.p.m. sodium silicate as silicon dioxide at a pH value of 8.9.

Sodium dichromate is an oxidizing agent and has been very successful in the elimination of organic odors from the wash water of air-conditioning systems, resulting in material savings in the cost of steam and power for both winter and summer operation by making possible the recirculation of a greater quantity of air. In most cases these savings have paid for the cost of treatment many times over.

Considerable information has been accumulated from actual corrosion experience had in these systems reported on in Table II to confirm the test findings. These inspections have been carried out by the author, owners of the equipment, and representatives of the manufacturers of the air-conditioning equipment. Table III contains a summary of the corrosion history of the systems in which the tests were run.

Obviously it is impossible to make complete inspection of large air-conditioning systems to examine all recesses inside of pipes, etc. The fact that sections easily visible failed from corrosion makes it possible to correlate reasonably closely the actual corrosion behavior of the systems with the predicted performances indicated in Table II by the test method. Actual experience has shown that dichromates have afforded the best protection.

DISCUSSION

Mr. P. R. Kosting[1] (*presented in written form*).—The author has shown another practical application of laboratory determined methods of retarding corrosion. In addition to the use of inorganic additions of known composition and also organic additions of unknown composition, referred to by Mr. Sterne, the so-called water-soluble oils have been found somewhat corrosion resisting when added to closed systems. It is wondered whether the use of soluble oil has been tried in these conditions. It is noted that a secondary consideration of any means of preventing corrosion is the elimination of odors which oxidizing agents effectively accomplish. Whether organic additions will be satisfactory from this point of view is questionable.

Mr. H. S. Rawdon.[2]—I should like to ask Mr. Sterne two questions. He has covered them in part in the last two sentences of the paper dealing with the correlation of the results of his tests with what is probably going on in the installation itself. First of all, does the recommended water treatment inhibit tendency towards electrolytic corrosion? Suppose there are two unlike metals in contact as part of the system; have any tests been made to show whether any particular treatment retards or inhibits the tendency toward electrolytic corrosion between those two metals? Secondly, we know that in "dead spaces" such as crevices, conditions are especially favorable to corrosion; this may result from a concentration cell such as an oxygen concentration cell or a concentration cell resulting from differences in the solution itself. We know definitely, however, that in crevices where sluggish conditions obtain, the situation is favorable towards corrosion. Have any experiments been carried out to show whether or not the recommended treatment of the water is favorable for inhibiting corrosion in the inaccessible dead spaces of the system where crevice corrosion would normally be expected to occur?

Mr. F. N. Speller.[3]—Mr. Sterne has applied one of the well-known principles of protection of metals, namely, by forming protective films.

Nearly all methods of corrosion prevention involve the building up of protective layers of some kind (solid or liquid) on the metal, whether that protective layer is formed from the metal itself or from the treatment of the environment, or the application of an artificial coating. Committee

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[1] Chemical Engineer, Watertown Arsenal, Watertown, Mass.

[2] Chief, Division of Metallurgy, National Bureau of Standards, Washington, D. C.

[3] Director, Department of Metallurgy and Research, National Tube Co., Pittsburgh, Pa.

A-5 on Corrosion of Iron and Steel is now considering some important work to determine the effect of corrosive water on various metals in air conditioning plants. The whole problem in connection with air conditioning plants requires systematic experimental study.

Several years ago we had a question of this kind in connection with the water used to cool transformers and found that a mixture of sodium dichromate, about 200 p.p.m., with 50 p.p.m. of caustic soda, gave the best results in Pittsburgh water. Mr. Sterne did not state whether he had tried that mixture or not. We found that practically complete elimination of corrosion was obtained by keeping chromate in the normal state; that is Na_2CrO_3. In regard to the influence of these inhibitors on the contact effect, we have studied that point in our research laboratory and find that the contact effect is very materially reduced by the use of chromate inhibitors. However, pitting results if the chromate treatment is insufficient.

MR. R. M. PALMER.[1]—Under the heading "Materials of Construction" it is stated that "galvanized steel [is used] for spray-head nipples and bronze for spray heads." In cases where preferred treatment No. 1 has been used, what amount of corrosion has been observed in connection with steel nipples? In Table II, under "Solution Used" (1) 42 per cent sodium dichromate, 6 per cent sodium hydroxide, (2) 40 per cent sodium hydroxide, 9 per cent sodium dichromate; which of the two formulas shown is the preferred formula? The U. S. Department of Agriculture, Meat Inspection Division, has recently ruled that chromate cannot be used for the treatment of water in air washers when the air washed later comes in contact with foodstuffs. Will you comment on this ruling?

MR. C. M. STERNE.[2]—In reply to Mr. Rawdon, when we find metals in direct contact, where they can be separated we usually put in insulating joints to break the couple. In regard to quiescent water we have had cases where the amount of fresh make-up water required was so great that it was impossible to carry proper concentrations of chemicals and heavy pitting occurred. In this situation we used treatment No. 4. To use anything else was too expensive so we advised our clients to change the type of condenser and to rearrange the system so a smaller quantity of make-up was needed and the corrosion problem was solved by using treatment No. 3.

In reply to Mr. Palmer on the question about galvanized spray nipples and bronze spray heads, we have observed slight corrosion in some cases and in others where treatments Nos. 1, 2 and 3 were used, practically none. We have seen very heavy corrosion on the nipples when treatment No. 4 was used. We usually advise painting the outside of the nipples since the threading operation destroys most of the galvanized coating.

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[1] Vice-President, Ferro-Nil Corp., New York City.

[2] Chief Engineer, Metropolitan Refining Co., Inc., Long Island City, N. Y.

That is where most of the corrosion will show up. In regard to treatment No. 1, both the solutions are used in conjunction; No. 1 consists of one solution to maintain the desired alkalinity and another to maintain the desired chromate concentration.

In regard to the ruling of the Department of Agriculture, about four years ago the fact was brought to our attention by the manufacturers of air conditioning equipment that in the case of food storage the air washers used salt brine as the spray medium and excessive corrosion was being encountered. In sodium chloride brine it is necessary to carry around 2000 to 2500 p.p.m. of sodium dichromate to obtain reasonable protection. There is always a carryover of salt through the eliminators to the conditioned spaces. We wrote the Department of Agriculture, Meat Inspection Division, pointing out these facts and stated that we would not do anything until we had received a ruling from them. They felt that in the case of brine solutions, therefore, chromate should not be used. I do not know of any ruling regarding the use of chromate in fresh water. We have no data on the contact effect of the various metals.

MR. PALMER.—The question I have raised in regard to these steel nipples, it seems to me, is the crux of this entire problem. I believe that the proper way to determine efficient protective methods for this problem of corrosion is to observe the equipment itself. Now in any air washer there are a large number of spray nozzles which are in turn, as Mr. Sterne has pointed out, connected with steel headers by means of steel nipples; in other words, if those steel nipples and headers corrode, you have one of your most serious problems of corrosion right here; that is where you are going to have high labor and high material cost for replacement and it seems to me that it is observations of that type that are far more important than observations of suspended samples in the water. In reply to my question about galvanized steel nipples, Mr. Sterne has stated that the corrosion "where treatments Nos. 1, 2 and 3 were used [was] practically none." It would appear then that some corrosion did take place when Mr. Sterne's preferred treatment was used, and painting the outside of the nipples is usually advised. I feel that that in itself indicates some concern about this problem, and yet he is treating this water for the prevention of corrosion, and perhaps in the most prolific place of corrosion, he is painting the steel nipples to protect them from corrosion.

MR. STERNE.—The particular air conditioning system referred to is in Chicago. I examined the system closely, looked at the nipples and spray nozzles and they are in fairly good shape. Still the water corroded the system and in a large air conditioning system it is impossible to tell where the corrosion attack will occur first. The best way is to protect everything possible by every means known to modern science, and even then corrosion attack is liable to take place.

(*Author's closure by letter*).—Subsequent to the presentation of the paper, the author inspected several large air conditioning systems using treatments Nos. 4 and 13. Particular note was taken of the condition of the nipples and in each case where they had not been painted a very heavy corrosion attack was found. The condition of the inside of various pipes carrying the treated water was found to be poor. Heavy rust barnacles spotted the surfaces; each barnacle that was brushed off covered a deep pit. There were no indications of any protective film or of any passivication of the metal surfaces.

The preferred reatments Nos. 1, 2 and 3 have been shown to give the best possible protection against corrosion of not only unpainted spray nipples, but throughout the entire system receiving treatment. No one could claim that in all cases, all corrosion of the ferrous metals could be completely stopped.

A test was run in both an air washer and a cooling tower using a soluble oil. .The foaming and carryover of the mixture was so great that the test had to be stopped after three hours' operation.

THE EFFECT OF COLD BENDING ON THE ELASTIC PROPERTIES OF HEAVY ($3\frac{1}{2}$ IN. THICK) STEEL PLATES

By C. H. Gibbons[1]

Synopsis

This paper reports the results of a series of experiments in which test specimens were taken from various parts of the plate both in the cold-bent condition and in the stress-relieved condition and stress-strain determinations were carefully made on each specimen. No conclusions are drawn as the paper is intended only as a presentation of little-known data on a complex and controversial subject.

Recently there arose the question of the effects of cold-working on steel plates such as are used in cracking-still service. The problem involved not only the effect on the physical properties of the plate as cold-worked but also the effect of stress-relieving or normalizing at various temperatures. The problem further involved determinations on plates of more than one chemical composition. The chemical composition from which the first series of test specimens were taken was: 0.17 per cent carbon, 0.51 per cent manganese, 0.015 per cent phosphorus, 0.024 per cent sulfur, 0.18 per cent silicon which meets the requirements for fire-box steel, A.S.M.E. Boiler Code, Section SI.

The large plate from which the section for test was cut was obtained by breaking down a 70 by 30-in. ingot weighing about 43,000 lb. to about 15 in. thickness, chipping all defects from the surface and rerolling to $3\frac{1}{2}$ in. thick. A section 90 by 24 by $3\frac{1}{2}$ in. was then gas-cut crosswise from the large plate and normalized at 1600 F. The plate edges were then machined to remove the effects of gas-cutting and the plate was ready for bending. A short section was cut from the end before bending and from this section were cut the test specimens which were to function as control specimens. All specimens were standard 0.505-in. diameter, 2-in. gage length specimens, threaded on ends.

The remainder (about 80 by 24 by $3\frac{1}{2}$ in.) was then bent cold in a bending roll having an 18-in. top roll and being only about 4 ft. long (to minimize the roll deflection and consequent "barreling" effect on the bent sheet) to a radius of $20\frac{1}{4}$ in. A section about 60 in. in length, forming an arc of nearly 180 deg., was bent (Fig. 1).

It is well known that the neutral surface of thick steel plate shifts in

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[1] Assistant Sales Manager, Southwark Division, Baldwin-Southwark Corp., Philadelphia, Pa.

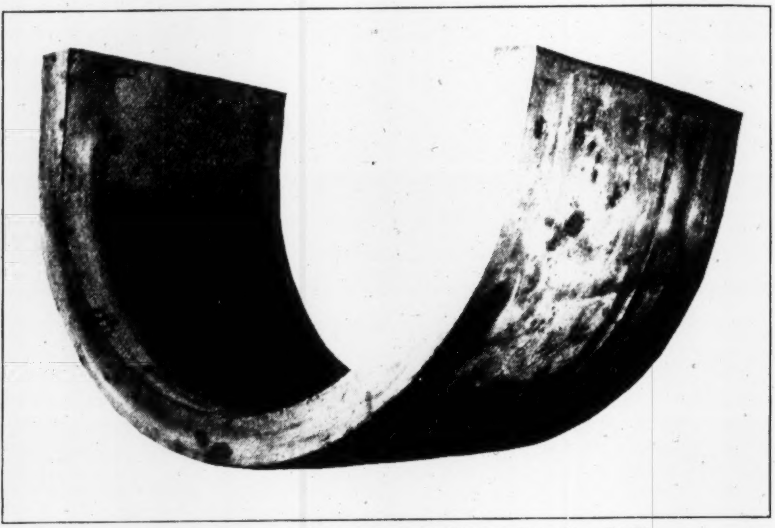


FIG. 1.—Showing Plate Bent Cold in a Bending Roll.

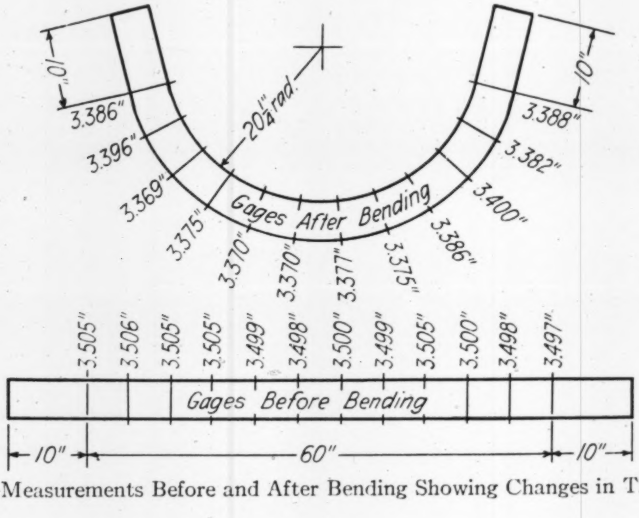

10"
3.386"
3.396"
3.369"
3.375"
3.370"
3.370"
3.377"
3.375"
3.386"
3.400"
3.382"
3.388"
10"
20¼" rad.
Gages After Bending
3.505"
3.506"
3.505"
3.505"
3.499"
3.498"
3.500"
3.499"
3.505"
3.500"
3.498"
3.497"
Gages Before Bending
10"
60"
10"

FIG. 2.—Measurements Before and After Bending Showing Changes in Thickness.

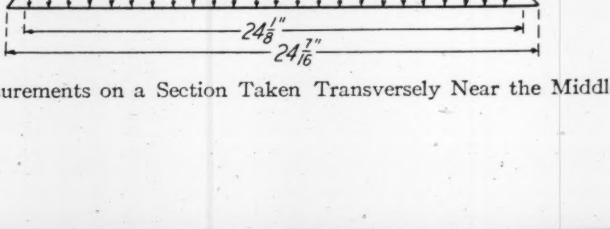

3.365"
3.415"
3.448"
3.473"
3.486"
3.493"
3.498"
3.493"
3.493"
3.499"
3.503"
3.498"
3.500"
3.501"
3.499"
3.505"
3.499"
3.506"
3.517"
3.512"
3.501"
3.459"
3.416"
3.337"
3/32" Out of Flat
24 1/8"
24 7/16"

FIG. 3.—Measurements on a Section Taken Transversely Near the Middle of Rolling.

bending towards the compression side—that is, that the extension in tension is greater than the shortening in compression. In order to secure quantitative values on this phenomenon, parallel lines 1 in. apart were scribed across both edges of the plate before bending. After bending, the distances were measured both on the inside and on the outside with the result that the neutral surface was $\frac{1}{8}$ in. inside the geometric mid-surface—that is, $1\frac{5}{8}$ in. from the inside cylindrical surface. Further, the average extension of the outside fiber had been 0.088 in. per inch or 8.8 per cent while the average shortening on the inside had been 0.076 in. per inch or 7.6 per cent. Prior to bending, also, the plate edge had been calipered at intervals.

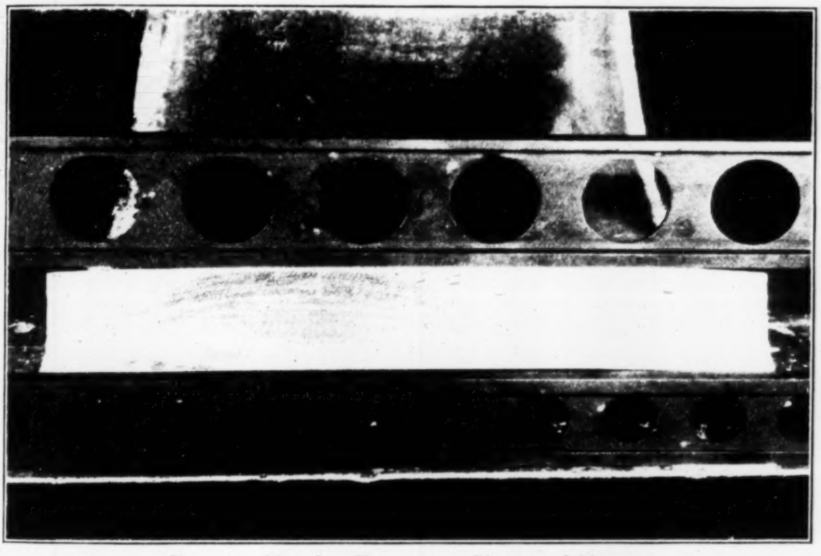


FIG. 4.—Showing Transverse Change of Shape.

After bending, similar measurements at the same places indicated that the plate had decreased in thickness at the edges, the greatest decrease being at the middle of the rolling of the cylinder (Fig. 2).

In order to discover the nature of this decrease in thickness across the width of the plate, a section transversely was taken near the middle of rolling and measurements taken (Fig. 3). It should be noted that the thinning of the plate is confined to the end 4 to 6 in. while the middle portion of the section has the same thickness as the original plate. The upper side of the section of Fig. 3 is the inside cylindrical surface. Straightedges show (Fig. 4) that there has been an over-all transverse change of shape, the amount of clearance between the straightedge and the plate surface at the ends being $\frac{3}{32}$ in. while there has been some "bowing" of the outside surface transversely as can be seen in the figure. This action has not been

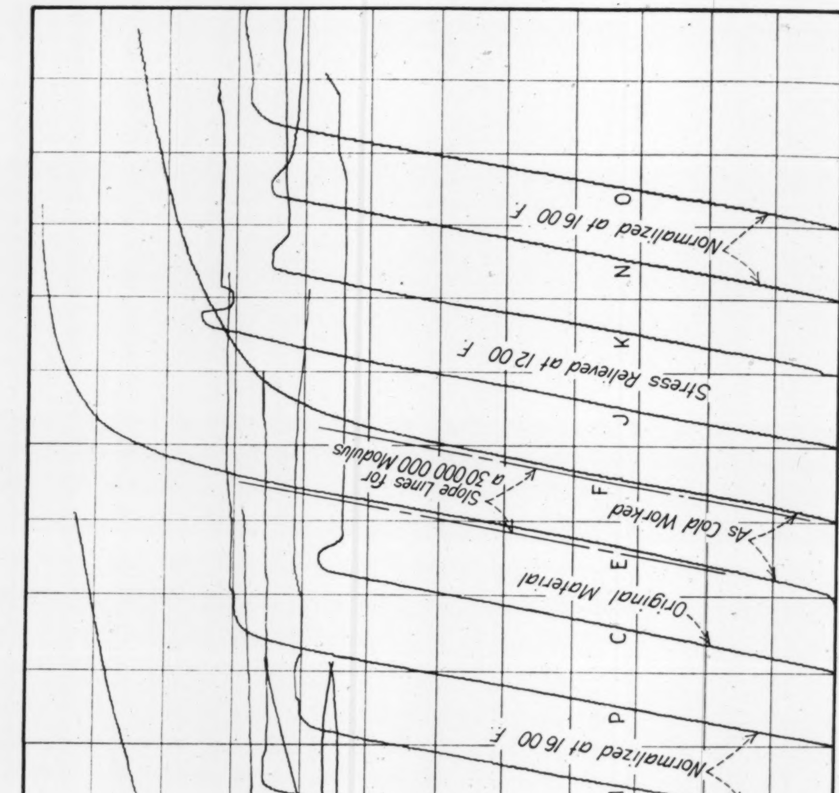


FIG. 5.—Stress-Strain Curves for Specimens Taken from Different Positions in the Section.

explained in a thoroughly satisfactory manner as it is opposite to the usual change in plate shape due to roll deflection. It is possible that there was not sufficient roll deflection over the short width of plate involved to show the characteristic "barreling" effect common on wider and thinner plates, but there was enough to relieve somewhat the middle portion of the plate width, concentrating the loads on the end portions (4 to 6 in. at each end) and deforming them by a transverse plastic deformation.

All of this, however, is more or less incidental to the main purpose of the investigation, which was to determine the effect of cold-working (bending) on the physical properties of the plate.

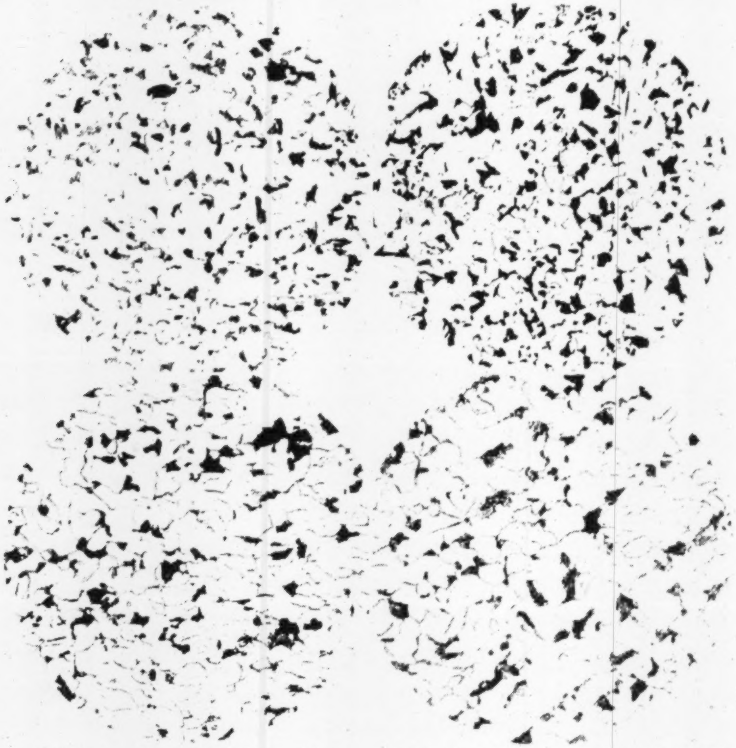
As control elements, specimens cut from the 24 by 10 by $3\frac{1}{2}$-in. section, previously mentioned, from positions as shown in Fig. 5 were prepared. The specimen A_2 was taken from a position near the surface and in the direction of proposed bending. Similarly specimen B_1 was taken from a position midway between the outer surface and the neutral surface and parallel to the axis of specimen A_2. Also specimen C at right-angles to specimens A_2 and B_1 and halfway between the outer surface and the neutral surface was prepared. As was to be expected, the stress-strain graphs showed the characteristic sharp knee of low-carbon annealed steel (Fig. 5, specimens A_2, B_1, and C).

From a quarter section of the rolled half cylinder, specimens D, E, F and G were cut from position indicated in Fig. 5. In each case the specimen was cut from a position midway between the surface (inside or outside) and the neutral surface. Specimens D and F were cut from the compression side in the direction of bending and at right-angles thereto, respectively, as indicated, and specimens E and G from the tension side in the direction of bending and at right-angles thereto, respectively. Because of the similarity of the graphs from specimens D and G and those from specimens E and F they have been grouped to bring them into juxtaposition. It will be recognized that the cold-working strains (and stresses) in specimens D and G were those of compression along their longitudinal axes. Similarly specimens E and F sustained tension strains. A close examination will reveal the similarity of the graphs in the elastic range and the truth of the statement. In like manner specimens H and L; J and K; M and P; and N and O have been placed side by side.

It is obvious that decided changes have occurred in the physical properties of the material. Not only has the shape of the graph changed radically but also the slope (which indicates the modulus of elasticity) has changed somewhat and there is no longer a definitive straight line which characterizes the group from the normalized specimens. Instead we find curves of long radius, but obviously curves, nevertheless. These curves indicate that there is no longer a constant modulus of elasticity within a proportional limit—in fact, there is no longer a proportional limit, using the commonly accepted definition. Specimens H, J, K and L were taken from positions corresponding to specimens D, E, F and G, respectively, but from a quarter section of the half cylinder which had been stress relieved at 1200 F. The graphs indicate that this treatment has re-established stress-strain relations quite similar to those of the original material.

But to check the results still further, another quarter section was normalized at 1600 F. for $3\frac{1}{2}$ hr. and air-cooled. Specimens M, N, O and P were then cut from positions corresponding respectively to positions D, E, F and G and graphs made. Comparison between this latter group and the original specimens A_2, B_1 and C provide an interesting study.

(*a*) Normalized edge. (*b*) Normalized middle.



(*c*) Tension side, "as bent" condition. (*d*) Tension side, "stress-relieved" condition.

FIG. 6.—Photomicrographs of Portions of the Sectional Area of the Original Plate.

Figure 6 (*a*) and (*b*) show photomicrographs of portions of the sectional area of the original plate from the edge and middle of section respectively. Figure 6 (*c*) and (*d*) show photomicrographs from the tension side of the "as bent" section and from the tension side of the "stress-relieved" (1200 F.) section, respectively. Similar photomicrographs from the normalized section showed still further refinement of grain structure.

Tests on 0.27 per cent Carbon Steel:

Later a series of tests was run on steel plate of different composition but having the same thickness, being bent to the same radius and having specimens cut from corresponding positions. The chemical composition was: 0.27 per cent carbon, 0.74 per cent manganese, 0.18 per cent silicon, 0.04 per cent, maximum, sulfur and 0.04 per cent, maximum, phosphorus.

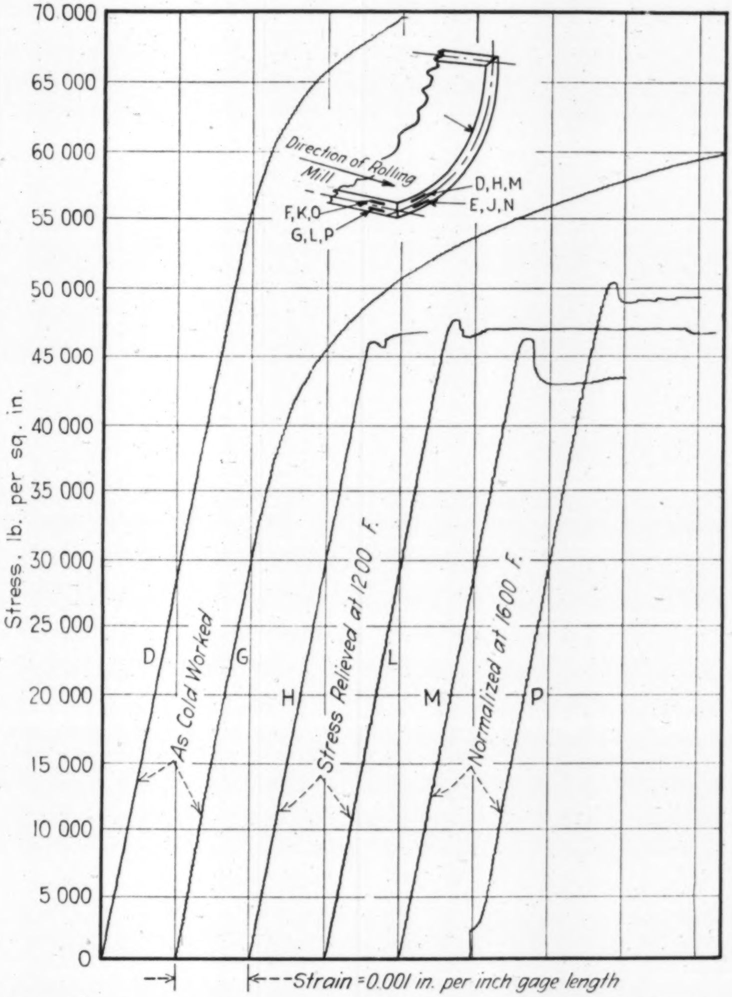


FIG. 7.—Stress-Strain Curves for 0.27 per cent Carbon Steel Plate.

A group of stress-strain curves from this steel is shown in Fig. 7. No graphs from material of the unworked plate were prepared as our experience with the first series had indicated that results from the "normalized at 1600 F." specimens would show stress-strain characteristics quite similar to those of the original material.

The tests on these specimens from the cold-worked plate indicated much straighter graphs in the elastic range than had been the case with the 0.17 per cent carbon steel. Modulus of elasticity and proportional limit (as usually defined) are fairly definite—a statement which could not be made of the 0.17 per cent carbon steel. Whether this is significant shall have to await further investigation.

Table I summarizes the data collected in both series of tests.

TABLE I.—DATA IN RESPECT TO SPECIMENS TESTED.

Specimen	Type of Cold Work[a]	Previous History	Position of Specimen in Respect to Mill Rolling		Tensile Strength, lb. per sq. in.	Yield Strength (0.2 per cent), lb. per sq. in.	Johnson's Limit, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
			Crosswise	Lengthwise					
0.17 PER CENT CARBON STEEL									
A_1	.....	1600 F.	x	....	62 500	....	37 000	38	36
A_2	.....	1600 F.	x	....	60 500	37 250	38 000	35	28.5
B_1	.....	1600 F.	x	....	61 450	38 250	41 000	40	38
D	C	Cold work	x	....	63 300	38 250	25 000	35	34
G	Cp	Cold work	....	x	66 300	52 500	43 000	25	25
H	C	1200 F.	x	....	61 500	43 750	45 000	34	35
L	Cp	1200 F.	....	x	61 500	42 750	42 000	35	32
M	C	1600 F.	x	....	63 500	40 500	38 500	38	52.5
P	Cp	1600 F.	....	x	62 000	45 500	43 500	33.5	58.6
C	.....	1600 F.	....	x	61 000	36 750	38 750	38.5	....
E	T	Cold work	x	....	67 300	58 500	50 000	25	27
F	Tp	Cold work	....	x	64 600	48 750	37 500	26	31
J	T	1200 F.	x	....	63 500	46 250	46 500	33.5	32
K	Tp	1200 F.	....	x	61 200	41 500	42 500	35	32
N	T	1600 F.	x	....	63 500	40 500	42 500	34	54.1
O	Tp	1600 F.	....	x	62 500	44 500	42 750	35.5	63.3
0.27 PER CENT CARBON STEEL									
D	C	Cold work	x	....	79 100	70 000	59 000	23	51
G	Cp	Cold work	....	x	75 900	53 500	40 500	27.5	54.5
H	C	1200 F.	x	....	71 750	....	46 000	37	56.5
L	Cp	1200 F.	....	x	72 250	47 000	47 000	33.5	56.5
M	C	1600 F.	x	....	74 750	....	46 000	35.5	57
P	Cp	1600 F.	....	x	73 500	....	49 750	28	56
E	T	Cold work	x	....	74 900	40 750	25 750	30	54
F	Tp	Cold work	....	x	76 250	48 750	33 500	29	54.5
J	T	1200 F.	x	....	72 050	46 250	46 000	35	56
K	Tp	1200 F.	....	x	72 400	46 500	34 250	33.5	57
N	T	1600 F.	x	....	77 150	43 500	47 000	26	39
O	Tp	1600 F.	....	x	74 750	44 000	43 000	35	57.8

[a] Cp indicates that cold work has been done by compression but the ratio Cp/C is of the order (in the elastic range, at least) of Poisson's ratio. In like manner Tp/T indicates a similar ratio in tension.

The account of this study is not intended to supply the last word on the subject of the effect on the physical properties of heavy steel plate of cold bending. It is hoped that it may add data of value to those interested in matters of this kind and to metallurgists in general.

Acknowledgment.—The author wishes to acknowledge with thanks the indispensable assistance rendered by a prominent steel company and to their assistant metallurgist particularly, in the supply of the steel and the data as to the case history of same.

THE HARDNESS TESTING OF LIGHT METALS AND ALLOYS

BY R. L. TEMPLIN[1]

SYNOPSIS

One of the most used tests in the commercial inspection of metals is the hardness test, of one form or another. The success attained in applying the hardness tests to ferrous metal products has quite naturally suggested similar uses for non-ferrous products.

A critical review of the factors affecting the more common hardness tests, however, reveals effects of different magnitude in some important instances, which would seem to mitigate seriously against the satisfactory use of the hardness tests for many of the light alloys. The presence of the oxide films on aluminum alloys, and the alloys coated with relatively thin layers of other different alloys are examples of the more conspicuous difficulties.

The effects of other factors such as load-penetrator-time relationships, anvil or support, size and preparation of specimen are quantitatively quite different than in the case of ferrous metals. Likewise the relationships between different kinds of hardness and between hardness and the other static properties of the light alloys are quantitatively different from those obtained in ferrous metals.

With these facts in mind the application of the hardness tests to general classes of commercial light-alloy products is discussed.

One of the most used tests in the commercial inspection of metals is the hardness test, of one type or another. The hardness tests most widely used in this country are the Rockwell, scleroscope and Brinell. The success attained in applying these hardness tests to ferrous metal products has quite naturally suggested similar uses in the non-ferrous product fields.

In the non-ferrous fields, however, the metals are generally softer, with the result that certain modifications in the test conditions must be made if satisfactory results are to be expected. These changes in test procedure usually involve lower loads on the penetrators, or larger penetrators, or both, in the case of the Rockwell and Brinell tests; and larger radius of striking face of the hammer (lower contact pressure) in the case of the scleroscope. These more obvious differences in conditions make it quite difficult to compare hardness values of ferrous and non-ferrous metals when using the prevailing standard methods.

In the application of these hardness tests to the light alloys of the non-ferrous metals, still other difficulties have been encountered, which will be discussed in more detail, that make comparisons of hardness values for different light-metal products quite unsatisfactory. In some specific instances, however, the hardness tests can be and are very useful in controlling the uniformity of certain operations applied to some of the light-metal products.

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A better understanding of the limitations of the hardness tests will obtain if a study is made of the quantitative effects of certain factors which affect the hardness values determined.

TABLE I.—SHOWING EFFECTS OF DIFFERENT LOAD AND BALL COMBINATIONS, DURATION OF LOAD AND LUBRICATION OF PENETRATOR ON THE BRINELL HARDNESS VALUES FOR SOME WROUGHT ALUMINUM ALLOYS.

All specimens 1 in. thick.

Material	Time, sec.	12.61-kg. Load, $\frac{1}{16}$-in. (1.59-mm.) Ball	500-kg. Load, 10-mm. Ball	1000-kg. Load, $\frac{9}{16}$-in. (14.3-mm.) Ball	500-kg. Load, 10-mm. Ball[a]					
					Acetone Cleaned		Machine Oil Lubricated		Gredag No.89 Lubricated	
					A	B	A	B	A	B
2S-O	15	26.0	24.0	24.8	24.9	25.3	24.5	25.1	24.9	25.1
	30	26.2	24.1	24.8						
	60	26.5	24.0	24.8						
	120	26.9	23.8	24.4						
Average		26.4	24.0	24.7						
2S-H	15	45.3	41.0	43.0	42.5	44.0	42.0	44.0	43.0	45.0
	30	44.5	41.0	43.0						
	60	42.9	40.0	43.4						
	120	42.2	42.0	42.9						
Average		43.7	41.0	43.1						
3S-O	15	31.6	28.6	29.0	28.4	29.3	29.3	29.7	29.3	30.0
	30	31.7	28.9	29.4						
	60	32.0	27.9	29.4						
	120	31.7	28.4	28.8						
Average		31.8	28.5	29.2						
3S-H	15	53.4	54.0	55.3	51.0	54.0	54.0	54.0	54.0	54.0
	30	52.5	52.0	55.1						
	60	54.5	52.0	54.8						
	120	53.8	54.0	54.1						
Average		53.6	53.0	54.8						
17S-T	15	109.4	106.0	112.0	106.5	106.5	106.0	107.0	106.0	105.0
	30	107.8	105.0	110.0						
	60	104.0	106.0	109.0						
	120	104.9	104.0	110.0						
Average		106.5	105.3	110.3						

[a] A = Regular ball. B = Hultgren etched ball.

THE BRINELL TEST

The Brinell hardness of light non-ferrous metals is defined as the ratio of a given load (in kilograms), applied to a hard metal ball of given size for a given time, to the spherical area (in square millimeters) of the recovered impression made in the test specimen. The basic conditions generally recognized for the test require the use of a 500-kg. load on a 10-mm. diameter steel ball for 30 sec. Both analysis and experiment have demonstrated that departures from this load and ball size can be made with quite satisfactory results if the load used is maintained equal to five times the square of the diameter of the ball used. In commercial testing balls from $\frac{1}{16}$ to $\frac{9}{16}$ in. in diameter are used.

Some Brinell hardness values for various ball and load combinations are given in Table I for certain wrought aluminum alloys representing the hardness range from 24 to 110. In the case of the $\frac{9}{16}$-in. ball the load used was not strictly $5D^2$, yet the hardness values obtained were quite comparable to the others. The data also show no differences in hardness for the variation in time of load application from 15 to 120 sec. In the case of wrought pure magnesium, however, the duration of load has a rather marked effect up to about 2 min., with much less effect beyond that time. It would seem necessary, therefore, in testing such material, to use a duration of load of not less than 2 min. for satisfactory results.

The etched Hultgren balls are sometimes used on brightly finished products to assist in reading impression diameters. Little if any attention is given to the question of ball lubrication in testing light metals and the results given in Table I would appear to justify this neglect. No differences that can be ascribed to lubrication are indicated between results obtained with etched Hultgren balls (dry) and polished steel balls lubricated with heavy colloidal graphite grease.

For Brinell hardness tests made using the 500-kg. load, 10-mm. ball combination, the depths of impressions will vary from 0.0313 in. for a Brinell value of 20, to 0.0063 in. for a value of 100. The depths of the impressions made by other sizes of balls under the proper loads will be directly proportional to the diameters of the balls.

The Society's Standard Methods of Brinell Hardness Testing of Metallic Materials (E 10 – 27)[1] state that the thickness of the specimen should be not less than ten times the depth of the Brinell impression, and according to Moore[2] the depth of impression should not exceed one-seventh the thickness of the specimen if comparable values are expected. The less conservative of these limitations would mean a minimum thickness of specimen of 0.219 in. when its Brinell value is 20, or a minimum thickness of 0.042 in. when its Brinell value is 100.

No change in the Brinell value of about 24, for annealed commercially pure wrought aluminum (2S-O), is found when using the 500-kg. load, 10-mm. ball combination, until specimens somewhat less than $\frac{1}{16}$ in. are tested as shown in Table II. Again, this set of test conditions can be used without change in hardness values, on the harder alloys having Brinell

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[1] 1933 Book of A.S.T.M. Standards, Part I, p. 942.

[2] Harold Moore, "Investigations in the Brinell Method of Determining Hardness," Fifth Congress, Internat. Assn. Testing Mats., II₁ (1909).

TABLE II.—HARDNESS VALUES FOR VARIOUS WROUGHT ALUMINUM ALLOYS; PLAIN AND "ALCLAD"; IN DIFFERENT THICKNESSES.

Thickness, in.	Brinell Hardness		Vickers Hardness		Rockwell Hardness[a]		Scleroscope Hardness, magnifier hammer
	12.61-kg. Load, $\frac{1}{16}$-in. (1.59-mm.) Ball	500-kg. Load, 10-mm. Ball	5-kg. Load	15-kg. Load	"E" Scale	"S" Scale	
2S-O							
$\frac{1}{64}$	24.7	19.6	22.2	21.9	−29.0	37.0	7.0
$\frac{1}{32}$	24.1	21.5	21.1	20.7	−31.0	37.0	7.0
$\frac{1}{16}$	23.4	23.1	22.5	21.4	−38.0	36.0	8.0
$\frac{1}{8}$	23.2	21.9	21.9	21.6	−34.0	33.0	8.0
$\frac{1}{4}$	24.5	24.5	24.7	23.6	−15.0	44.0	8.0
17S-T							
$\frac{1}{64}$	96.0	77.7	113.2	111.9	99.0	112.0	27.5
$\frac{1}{32}$	98.7	105.0	115.2	117.1	95.5	110.0	29.0
$\frac{1}{16}$	98.7	103.3	111.2	115.8	96.5	110.5	31.0
$\frac{1}{8}$	102.3	101.5	110.2	114.1	97.5	111.0	33.5
$\frac{1}{4}$	104.9	111.0	114.1	117.1	98.5	113.0	35.5
ALCLAD 17S-T							
$\frac{1}{64}$	78.1	62.0	92.0	101.9	91.5	108.5	26.0
$\frac{1}{32}$	57.6	90.0	65.5	87.6	92.0	109.0	27.0
$\frac{1}{16}$	44.3	83.0	47.6	71.3	92.5	108.5	27.5
$\frac{1}{8}$	30.2	73.0	29.1	41.5	87.0	95.5	22.0
$\frac{1}{4}$	22.4	51.4	22.2	23.9	50.0	80.0	11.5
17S-RT							
$\frac{1}{64}$	105.8	86.5	121.5	123.3	100.5	113.0	29.5
$\frac{1}{32}$	109.7	111.3	120.5	124.0	99.5	111.0	34.0
$\frac{1}{16}$	117.0	111.8	123.8	126,6	101.5	114.0	36.0
$\frac{1}{8}$	122.1	121.0	127.2	137.4	103.0	115.0	42.0
$\frac{1}{4}$	120.1	121.0	129.6	128.7	103.0	114.5	40.0
ALCLAD 17S-RT							
$\frac{1}{64}$	74.9	91.0	93.4	108.0	98.0	111.0	28.0
$\frac{1}{32}$	68.1	100.0	80.0	92.8	96.5	110.0	31.5
$\frac{1}{16}$	36.5	86.0	38.8	53.1	95.5	109.0	28.0
$\frac{1}{8}$	28.4	75.5	29.5	35.7	87.0	102.0	20.5
$\frac{1}{4}$	21.8	53.3	24.0	25.2	52.0	81.5	11.0
24S-T							
$\frac{1}{64}$	107.3	95.3	133.3	132.2	100.0	114.5	29.5
$\frac{1}{32}$	108.6	121.8	135.9	134.4	100.0	113.0	34.0
$\frac{1}{16}$	108.5	116 3	128.4	130.1	100.0	114.0	36.0
$\frac{1}{8}$	117.3	116.5	127.2	128.0	103.0	114.0	39.0
$\frac{1}{4}$	118.5	118.5	127.2	130.9	102.0	114.0	38.0
ALCLAD 24S-T							
$\frac{1}{64}$	89.3	89.0	113.2	113.0	98.0	112.0	28.0
$\frac{1}{32}$	64.2	97.5	76.1	89.9	97.0	111.0	30.5
$\frac{1}{16}$	43.5	91.0	45.8	69.8	97.0	110.5	30.0
$\frac{1}{8}$	29.1	79.8	29.2	37.4	88.0	102.0	21.0
$\frac{1}{4}$	22.2	55.3	22.2	24.4	54.5	84.0	11.5
24S-RT							
$\frac{1}{64}$	109.4	95.5	135.9	134.4	100.5	115.0	30.0
$\frac{1}{32}$	118.0	129.2	142.6	142.9	101.0	115.0	37.0
$\frac{1}{16}$	118.0	124.7	142.6	142.1	103.5	116.0	40.5
$\frac{1}{8}$	123.9	124.6	137.2	137.4	104.0	116.0	42.0
$\frac{1}{4}$	122.1	128.0	137.2	136.6	104.0	116.0	39.0
ALCLAD 24S-RT							
$\frac{1}{64}$	91.6	106.0	101.4	116.4	100.5	113.0	29.5
$\frac{1}{32}$	72.4	111.0	76.2	94.1	100.0	113.5	37.0
$\frac{1}{16}$	40.5	97.5	43.6	61.6	99.0	111.0	32.0
$\frac{1}{8}$	28.6	82.5	28.9	39.1	88.5	102.5	22.0
$\frac{1}{4}$	23.7	56.3	26.0	25.1	55.0	82.0	11.0

[a] "E" scale—100-kg. major load, $\frac{1}{8}$-in. ball (red or "B" scale).
"S" scale—60-kg. major load, $\frac{1}{8}$-in. ball (red or "B" scale).

values around 100, if the specimens are not less than $\frac{1}{32}$ in. thick. It would appear, therefore, that for satisfactory results in the case of the wrought aluminum alloys, the thickness of Brinell specimens for the softer alloys needs to be but little, if any, more than twice the depth of impression, and for the harder alloys no more than five times the depth of impression.

In making static indentation hardness tests on light metals, no differences in results are noticeable when either hard or soft steel anvils or specimen supports are used, provided the specimens are of suitable thickness as previously indicated. When soft anvils of other metals are used, effects can be observed, but since such supports are not ordinarily used or considered in commercial testing they will not be discussed further herein.

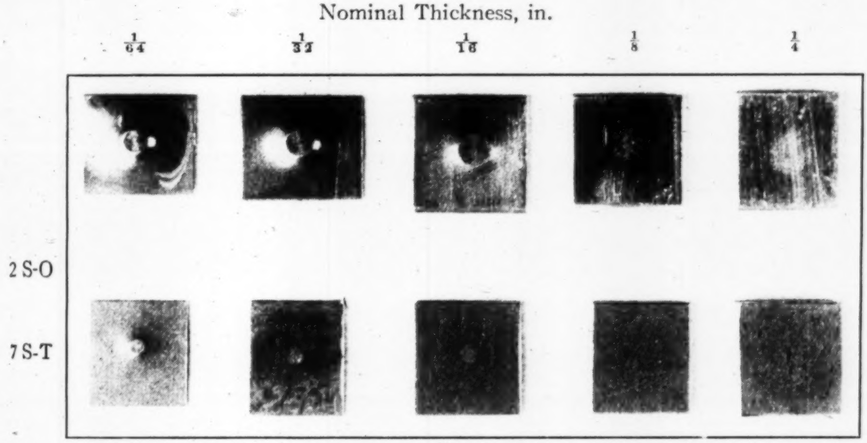

FIG. 1. —Marks Made by Brinell Impressions on Various Thicknesses of Soft and Hard, Wrought Aluminum Alloys.

Another rule, which appears in the standard methods for Brinell hardness tests of metals, states that when the side of the specimen opposite the impression shows distortion caused by the impression, the results are questionable. In the case of the light metals, however, considerable distortion can occur before there is any appreciable effect on the hardness values obtained. The specimens shown in Fig. 1 correspond to some of those for which Brinell data are given in Table II.

The Brinell hardness values obtained in tests of light metals may be, and often are, appreciably affected by the surface conditions of the specimens used. Many of the light-metal products such as common alloy sheet, bar and extruded shapes have smooth bright surfaces which require no additional treatment in order to make them suitable for hardness tests. Most sand castings and rough forgings, however, require additional surface preparation. For routine commercial inspection testing this is usually done by filing, scraping, or grinding. In some instances further finishing may be done using fine emery cloth followed by buffing. The alloys that are heat treated have oxide coatings which are somewhat thicker than those on the cold-worked common alloys; and some of the products have comparatively thick anodic coatings, purposely applied electrolytically to obtain better corrosion resistance or paint adherence, or both. In the case of such products it is difficult to obtain satisfactory hardness determinations because if they are tested without removing the coating, variations in results may be obtained depending upon the load-penetrator combination used. Removal of the coating involves damage to the product unless a separate coupon is provided. The variations in hardness of two materials with different amounts of oxide coating are shown in Table III. It may be noted that the greater variations occur when using the smaller penetrators and lighter loads. The type of Brinell impression obtained on anodically coated aluminum is shown by the enlarged photograph in Fig. 2. The radial cracks in the coating are quite typical.

TABLE III.—HARDNESS VALUES OBTAINED ON WROUGHT ALUMINUM ALLOYS WITH AND WITHOUT OXIDE COATINGS.

All specimens $\frac{1}{4}$ in. thick.

Material	Surface Condition	Brinell Hardness		Vickers Hardness		Rockwell Hardness[a]		Scleroscope Hardness, magnifier hammer
		12.61-kg. Load, $\frac{1}{16}$-in. (1.59-mm.) Ball	500-kg. Load, 10-mm. Ball	5-kg. Load	15-kg. Load	"E" Scale	"S" Scale	
2S, as rolled..	freshly machined	33.0	35.4	37.1	38.3	35	79	14
	original oxide coating	34.5	35.1	38.6	38.7	35	79	14
	0.0002-in. oxide coating	38.8	36.4	40.2	39.7	36	80	14
	0.0008-in. oxide coating	39.8	36.8	44.3	40.9	36	80	15
53S-T........	freshly machined	87.3	92.0	89.2	92.8	93	109	32
	original oxide coating	86.0	89.0	91.1	90.3	93	109	31
	0.0002-in. oxide coating	87.3	91.2	94.6	92.0	93	109	32
	0.0008-in. oxide coating	97.2	91.0	104.8	98.1	94	109	32

[a] "E" scale—100-kg. major load, $\frac{1}{8}$-in. ball (red or "B" scale).
"S" scale—60-kg. major load, $\frac{1}{8}$-in. ball (red or "B" scale).

In making the Brinell test it is usual practice to average the values obtained from two diameter measurements of the impression taken 90 deg. apart. In some instances, as shown in Fig. 3, Brinell, Rockwell and Vickers impressions are obtained which are appreciably out of shape. The distortions observed in practice may amount to as much as 15 per cent in the Brinell and Vickers values, yet such materials will exhibit but slight differences in their tensile properties in the various directions. In such instances any deductions concerning directional properties of the materials, based on the hardness tests, are likely to be quite misleading quantitatively.

The errors involved in making the Brinell test include a permissible testing machine load error of ±3 per cent, an error in ball diameter which is usually negligible but may affect the results by ±1 per cent, a diameter of impression measurement which is usually within ±2 per cent and in

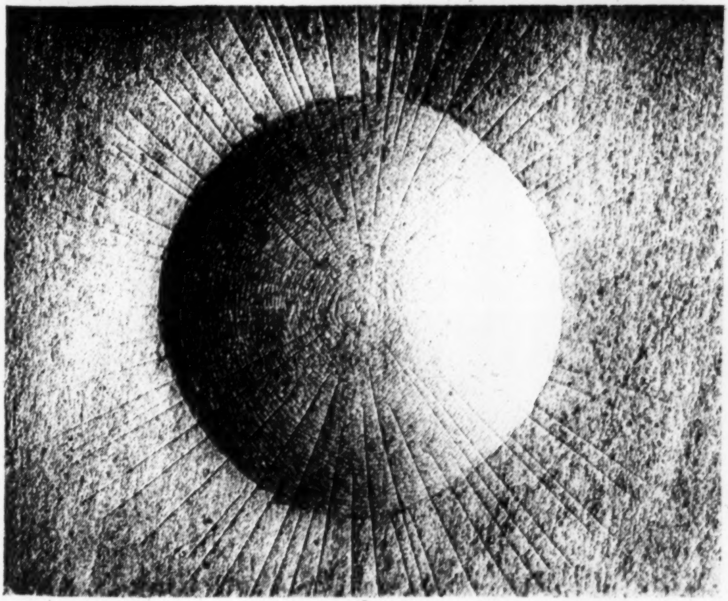

FIG. 2.—Enlarged Photograph of Brinell Impression Made on Anodically Coated Aluminum Alloy Sheet.

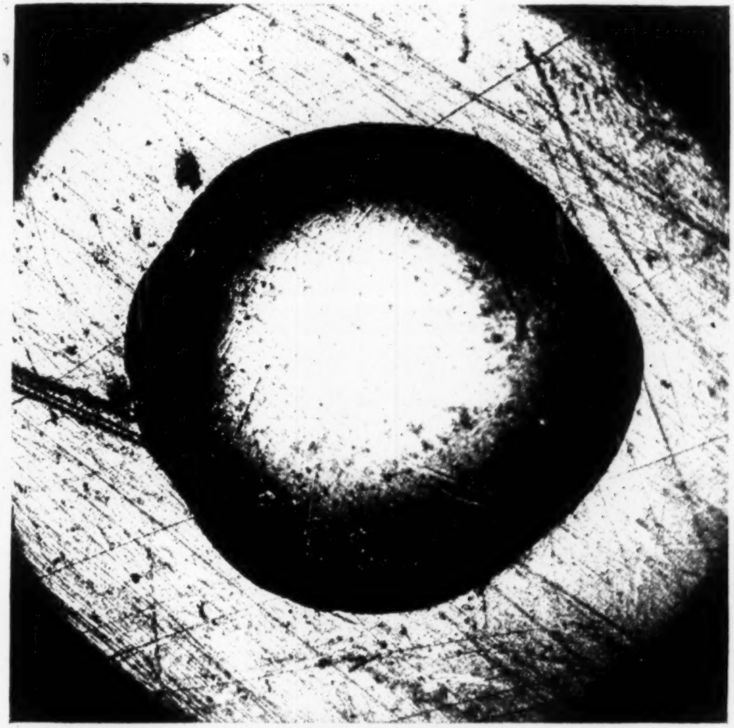

FIG. 3.—Enlarged Photograph of Distorted Brinell Impression as Obtained on Certain Aluminum Products.

routine commercial use, an error resulting from hasty manipulation of the test that probably amounts to ±2 per cent in hand-operated machines. Thus these errors, which are often additive, may total as much as 8 per cent but on the average will be within about ±5 per cent. It is only fair to point out, however, that comparisons of Brinell hardness results from different commercial laboratories, using the same test specimens, have shown differences of as much as 15 per cent.

When using the smaller ball and load combinations for the Brinell test, it is necessary to use a compound filar microscope for measuring diameters of impressions. Such a microscope should permit estimation of diameter to within ±0.002 mm. Under these conditions the making of Brinell hardness determinations is rather slow and expensive, with considerable opportunity for errors in manipulation.

THE ROCKWELL TEST

The Rockwell test may be defined essentially as the difference in depth between an initial indentation caused by a primary load (10 kg.) and a final indentation resulting from a larger secondary or major load, while the penetrator is held in the final indentation by the primary load. In commercial practice quite a variety of loads and penetrators are used. Those generally recognized for use in testing light metals include a major load of 100 kg. on a $\frac{1}{8}$-in. diameter steel ball with hardness values determined on the red scale of the dial (designated as the Rockwell "E" scale hardness); and a major load of 60 kg. on a $\frac{1}{8}$-in. steel ball using the same scale (designated as the Rockwell "S" scale hardness). The latter of these scales is not recognized in the Society's Tentative Methods of Rockwell Hardness Testing of Metallic Materials (E 18 – 33 T).[1] An approximate comparison of the hardness values obtained on light metals using these two sets of test conditions with the standard Brinell test values is given in Table IV. The relationships may be expressed by the following equations:

$$\text{B.H.N.} = \frac{3570}{130 - R_E}$$

and

$$\text{B.H.N.} = \frac{2460}{140 - R_S}$$

where B.H.N. = Brinell hardness,

R_E = Rockwell hardness, "E" scale, and

R_S = Rockwell hardness, "S" scale.

From what has been pointed out in discussing the Brinell test concerning limitations of specimen thickness and depth of impression, and the load - ball size conditions of the Rockwell "E" and "S" scales, it may be seen that these two Rockwell hardness scales will cover about the same range of thickness for light-metal products as that which may be satisfactorily tested with the different Brinell ball sizes, $\frac{1}{16}$ in. in diameter and larger.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 967 (1933); also 1934 Book of A.S.T.M. Tentative Standards, p. 1109.

The Rockwell test is more readily carried out than either the Brinell or Vickers and probably involves less personal equation. In attempting to use the test in the commercial inspection of light metals, however, considerable difficulties have been encountered. These include troubles in calibration and maintenance of machines, erroneous or insufficient designation of hardness values recorded, reading wrong scale on dial, and use of wrong load or penetrator in machines equipped for various combinations of these. A mistake frequently made in attempting to test thin specimens is to superimpose a number of thin specimens. This leads to erratic results. Near the lower ends of the Rockwell hardness scales suitable for use on light metals, quite large variations can be obtained in values, which have relatively little significance, if the tensile properties are used as an index of the quality of product. Near the upper ends of the scales the opposite is

TABLE IV.—COMPARISON OF BRINELL HARDNESS VALUES AND ROCKWELL "E" AND "S" SCALE VALUES FOR WROUGHT ALUMINUM ALLOYS.

Brinell Hardness[a]	Rockwell Hardness[b]		Brinell Hardness[a]	Rockwell Hardness[b]	
	"E" Scale	"S" Scale		"E" Scale	"S" Scale
14	..	−30	48	56	89
15	..	−20	49	57	90
16	..	−10	50	59	91
18	..	0	51	60	92
19	..	10	52	61	93
20	..	20	53	63	94
22	..	30	54	64	94
25	..	40	55	65	95
27	..	50	56	66	96
28	2	52	57	67	97
29	7	55	58	68	98
30	11	58	59	69	98
31	15	61	60	70	99
32	18	63	61	71	100
33	22	65	62	72	100
34	25	68	63	73	...
35	28	70	64	74	...
36	31	72	65	75	...
37	33	73	70	79	...
38	36	75	75	82	...
39	38	77	80	85	...
40	41	78	85	88	...
41	43	80	90	90	...
42	45	81	95	92	...
43	47	83	100	94	...
44	49	84	105	96	...
45	51	85	110	98	...
46	52	87	115	99	...
47	54	88	120	100	...

[a] Based on 500-kg. load, 10-mm. ball, 30 sec.

[b] "E" scale—100-kg. major load, $\frac{1}{8}$-in. ball (red or "B" scale).
"S" scale—60-kg. major load, $\frac{1}{8}$-in. ball (red or "B" scale).

true. This may be readily seen from the curves and data given in Fig. 4. As in the case of the use of the smaller Brinell balls, for satisfactory results the Rockwell test will generally require more careful surface preparation of the specimens than when using the larger balls in the Brinell test.

THE VICKERS TEST

In the Vickers hardness test the penetrator used is a square diamond pyramid having a total included angle of 136 deg. The values for hardness are obtained by dividing the load (in kilograms) by the surface area of the impression (in square millimeters). Throughout the range of loads generally used, the hardness values are constant, which offers a distinct advantage over the Brinell and Rockwell tests, and the loads can be varied to suit both the hardness and thickness of the specimens without change in penetrator. When using the smaller loads, from 1 to 15 kg., however, it is necessary to use the more accurate filar microscope as in the case of the "baby" Brinell test.

The limitations regarding specimen thickness are apparently somewhat less severe for the Vickers than for the Brinell test and therefore cause little trouble in tests of light metals. In practice the Vickers test can be made in about the same time as is required for the Brinell test. The impressions obtained are easier to measure because of their square shape, the diagonals only being measured. Distorted impressions, however, occur as in the Brinell test. On the whole the errors in the Vickers test are probably somewhat less than in the Brinell test. Some comparative Vickers hardness values are given in Tables II and III for some of the aluminum alloys.

THE SCLEROSCOPE TEST

In the Shore or scleroscope hardness test a small hammer weighing about 2.4 g. is dropped from a height of about 10 in. on the specimen, and the height of rebound is observed on an arbitrary scale of 140 divisions = 10 in. (model C or indicating type). Another form of the instrument (type D) uses a much heavier hammer which drops about $\frac{3}{4}$ in. In this case the hammer is caught by a suitable mechanism at its maximum height of rebound and the height recorded on a dial scale graduated into 140 divisions, so that readings are obtained which agree more or less with those obtained with the indicating or type C instrument. Both instruments are provided with hard steel anvils or specimen supports and a clamping arrangement to hold the specimens firmly in position while being tested.

In both types of the scleroscope it is necessary to use the magnifier hammer for satisfactory results on the light metals. These hammers may be furnished with either hardened steel or diamond points and in either case have larger radii of curvature than the "universal" diamond-pointed hammers. Unfortunately much trouble has been experienced in obtaining

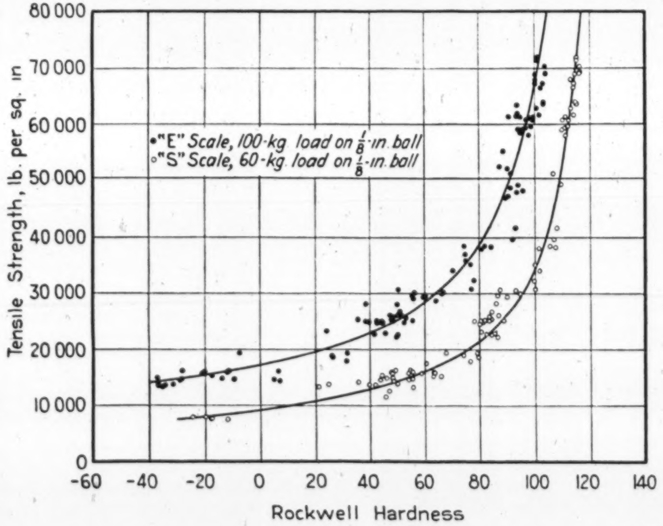

FIG. 4.—Tensile Strength - Rockwell Hardness Relationship for Wrought Aluminum Alloys.

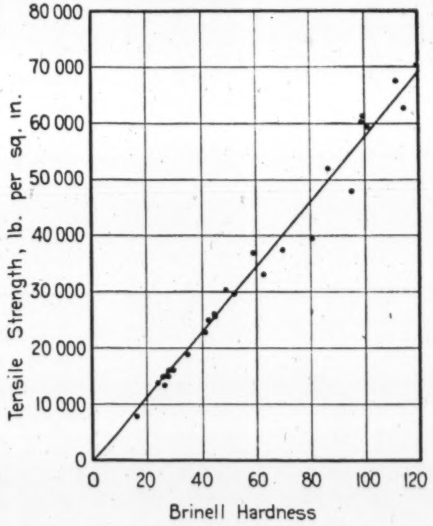

FIG. 5.—Tensile Strength - Brinell Hardness Relationship for Wrought Aluminum Alloys.

magnifier hammers that will check similar hammers in other instruments of the same type. In England, largely as the result of investigation by Genders,[1] a magnifier hammer for the indicating type of instrument has been adopted which apparently gives quite concordant results and can be rather easily maintained within the desired limits of accuracy. In this country, however, there are a large number of scleroscopes in use with magnifier hammers that give quite discordant results on the light metals, thus making it very difficult if not impossible to obtain satisfactory check results amongst the users of these instruments.

Limitations concerning thickness of specimen are somewhat more severe in the use of the scleroscope than with the other hardness tests discussed. From the data given in Table II, it may be seen that for both the softer and harder materials the scleroscope readings are affected when the specimens are less than about $\frac{1}{16}$ in. thick. With the scleroscope test, of course, no change in hammer size can be made, while in the static penetration tests the conditions can be altered to test satisfactorily appreciably thinner specimens. As in the case of the Rockwell test attempts are frequently made to test thin specimens with the scleroscope by superimposing a number of thin specimens on each other. Experience shows that a different hardness value is usually obtained with the addition of each piece. The personal errors involved in reading and manipulating the scleroscope are often large in the case of the indicating type.

In spite of the difficulties just noted, the scleroscope can be and is used to good advantage in certain routine inspection tests in controlling some fabrication operations. It has not been considered satisfactory, however, as a final test for acceptance or rejection of light-metal products and should not, therefore, be considered as a suitable specification test.

COMPARISON OF HARDNESS VALUES WITH EACH OTHER AND WITH THE TENSILE PROPERTIES

Many metals, especially in the wrought condition, exhibit a rather definite and usually simple relationship between their Brinell or Vickers hardness values and tensile strength. The wrought aluminum alloys are no exception to this as may be seen from Fig. 5. The points indicated are in most cases the average of a considerable number of determinations and represent the average values for various grades and tempers of commercial products. The tensile strength - Brinell relationship as shown by the straight line in Fig. 5 may be expressed by the simple equation:

$$\text{B.H.N.} = \frac{T}{575}$$

where B.H.N. = Brinell hardness number, and

T = tensile strength, lb. per sq. in.

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[1] R. Genders, "The Scleroscope Hardness Test. A New Form of Magnifier Hammer," *Journal*, Inst. Metals (British), Vol. XXIX, Part I, p. 445 (1923).

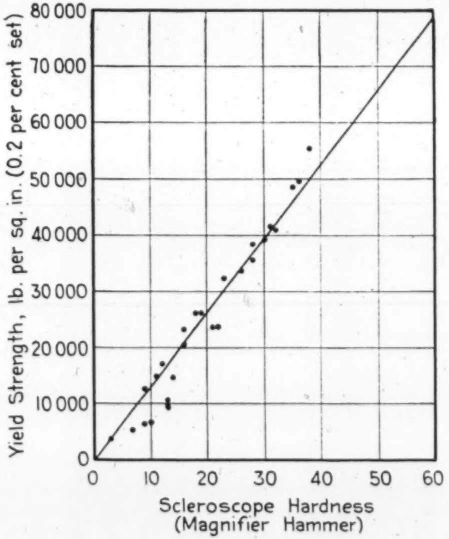

FIG. 6.—Yield Strength - Scleroscope Hardness Relationship for Wrought Aluminum Alloys.

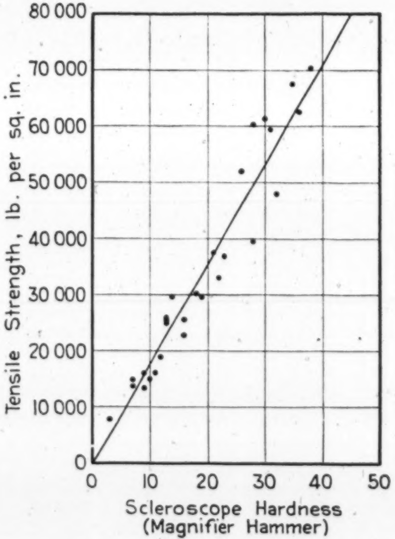

FIG. 7.—Tensile Strength - Scleroscope Hardness Relationship for Wrought Aluminum Alloys.

In giving this equation, however, it is appreciated that there is a definite minimum Brinell hardness number possible, corresponding to that obtained with an impression equal to the diameter of the ball used. This minimum value is not zero as indicated by the equation just given, but the errors resulting are negligible in most instances, and in the interest of simplification have been ignored.

Unfortunately the procedure too often followed is to try to determine the tensile strength of a product from a Brinell test and the relationship just indicated. In doing so the errors involved in the Brinell determinations are multiplied 575 times with the result that the values for tensile strength obtained by this procedure may be very much in error. On the other hand, if we know the tensile strength of the material, the Brinell hardness can be calculated from the equation given, with a very satisfactory degree of accuracy. A similar relationship for Vickers hardness and tensile strength can be worked out with the same pertinent limitations.

The relationship between Brinell and Rockwell hardness for aluminum alloys is shown in Table IV and the equations given previously. The comparison in this instance is rather complex and in practice generally results in the use of the table rather than the equations given. The relationship between tensile strength and Rockwell hardness is likewise rather complex as shown by the curves in Fig. 4. The equations for these curves are:

$$T = \frac{2{,}020{,}000}{(130 - \mathrm{R_E})}$$

and

$$T = \frac{1{,}320{,}000}{(140 - \mathrm{R_S})}$$

The constants in these equations are quite large and their numerical accuracy rather questionable. In general form, the equations are quite in agreement with those proposed by Petrenko.[1]

The scleroscope hardness values seem to correlate better with the yield strengths of the light metals, as shown in Fig. 6, than with the tensile strength values, although there is but little more scatter in the latter case as shown by Fig. 7. These relationships may be approximately represented by the following equations:

$$S = \frac{Y}{1320} \text{ and } S = \frac{T}{1775}$$

where S = scleroscope hardness (magnifier hammer),
Y = yield strength, 0.2 per cent set, lb. per sq. in., and
T = tensile strength, lb. per sq. in.

Here, as in the case of the Brinell relationship, it is quite unsatisfactory to attempt to determine the tensile strength or yield strength from a scleroscope value; but knowing the value of tensile strength or yield strength the scleroscope hardness can be estimated rather closely from the equations given.

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[1] S. N. Petrenko, "Relationship Between Rockwell and Brinell Numbers," National Bureau of Standards *Journal of Research*, Vol. 5, No. 1, July, 1930, p. 19.

HARDNESS OF COATED LIGHT METALS

In recent years, a new variety of highly corrosion-resistant, sandwich-type light-metal products has been used extensively in various commercial fields. In this country these products are marketed under the trade name "Alclad." Those first offered to the trade had a duralumin core with a thin coating of pure aluminum on the outer surface. These coatings were usually about 5 per cent of the total thickness of the product. Any attempt to obtain hardness tests on such products leads to quite unsatisfactory results as may be seen from a study of the data given in Table II. It would be quite difficult to remove the proper amount of coating from samples of different thicknesses in order to obtain satisfactory hardness values of the core metal, and as in the case of the anodically coated alloys such a procedure would damage the product seriously unless separate coupons were used.

In conclusion it may be pointed out that the Brinell test, especially the 1000-kg. load, $\frac{9}{16}$-in. ball combination, is quite extensively used as a 100 per cent final inspection test on aluminum pistons, made as forgings, sand castings, or permanent-mold castings. This test is also used as a check on the heat treatment of many other light-alloy products in both the wrought and cast condition. The Vickers test has so far been used to little extent in the light-metal field in this country. While both the Rockwell and scleroscope tests have been used to considerable extent in testing light-metal products, yet for reasons discussed their use in product specifications would appear quite unsatisfactory. In general, experience has shown the tension test to be quite preferable to any of the hardness tests, when the form and size of product permit of its use. From tension test values of light metals in the wrought condition, the various hardness values can be computed with satisfactory results, using the relationships given.

Many phases of the general subject of hardness testing of metals have been ably treated by O'Neill[1] and this reference is recommended for those interested in certain academic questions which may occur. The author wishes to acknowledge the assistance of Mr. C. R. Buckles in obtaining many of the test data given.

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[1] Hugh O'Neill, "The Hardness of Metals and Its Measurement," The Sherwood Press, Cleveland, Ohio (1934).

DISCUSSION

Mr. F. B. Fuller[1] (*presented in written form*).—Experience in hardness testing at Wright Field in general is in accord with the factors reported in Mr. Templin's paper.

The study of thin gage sheet and of small specimens from failed parts has necessitated the adaptation of other methods to obtain results comparable to those with the standard Brinell. The application of the Vickers hardness testing machine using steel balls of 1 and 2-mm. diameter with a load in kilograms of five times the square of the diameter of the ball for non-ferrous alloys, as recommended in the Society's standard procedure has proven very satisfactory and convenient. The data noted in the accompanying Table I are comparable to those from the standard Brinell (500-kg. load and 10-mm. ball) and are also in agreement with those given in the paper. The data obtained by Templin and at Wright Field with the Vickers test with a load greater than $5D^2$ and with the diamond pyramid indenter are somewhat higher than the standard Brinell for the hard non-ferrous alloys but are comparable for the softer or annealed alloys. The results from the Vickers, therefore, coordinate closely with the Brinell test and the testing involves less of the personal equation. The results obtained with different loads on the same type of penetrator are approx-

Table I.—Hardness Values for Aluminum Alloys.

Material	Condition	Thickness, in.	Brinell Hardness, 500-kg. Load, 10-mm. Ball	Vickers Hardness, Steel Balls			Vickers Hardness, Diamond Indenter, 30-kg. Load
				5-kg. Load, 1-mm. Ball	20-kg. Load, 2-mm. Ball	20-kg. Load, 1-mm. Ball	
Aluminum alloy propeller stock........	Wrought......	......	......	106	113	......	......
		0.500	111	111	115	......	......
Aluminum magnesium alloy.............	Cast bar......	......	......	69.5	72	......	......
		0.500	72	73	74	......	......
Aluminum alloy.......	Rolled bar....	......	101	105	103	......	115
		0.500	102	109	105	......	117
Aluminum alloy.......	Cast bar......	......	......	53	60	......	64.1
		0.500	55	63	65	......	64.6
17S-T...............	Quenched sheet	......	......	......	72.3	......	78.6
		0.032	......	......	74.7	......	79.6
17S-T...............	Aged sheet....	......	......	......	105.3	......	116
		0.032	......	......	107.2	......	119
24S-T...............	Sheet.........	......	118	116.5	119	136	......
		0.092	119	118.0	123	136	......

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[1] Associate Materials Testing Engineer, U. S. Air Corps, Wright Field, Dayton, Ohio.

imately directly proportional. So far the only difficulty of major importance with the Vickers test has been to obtain, domestically, diamonds ground to the same degree of precision as noted in the one obtained from England.

Since it has been the policy at Wright Field to use another type of test or load in lieu of the 500-kg. load on the 10-mm. ball whenever the effect of distortion was found on the opposite face, the results given in the paper along these lines are worthy of note.

The effect of surface conditions, such as the use of a softer layer of outside material and of oxide coatings, is a serious factor, especially when using a machine in which the diameter of the impressions are read. Brinell data on non-ferrous hard-rolled sheet with and without a softer covering are in accord with those in the paper.

In the hard-rolled non-ferrous alloys the Brinell hardness has given a good approximation of the tensile strength, but in the cast alloys it is of value only for indicating satisfactory heat treatment.

MR. C. E. TOPPING[1] *(presented in written form)*.—Our experience in the hardness testing of light metals and alloys has been primarily with a series of magnesium-base alloys. We have found the Rockwell hardness tester, using the Rockwell "E" scale, to be very satisfactory in our work on magnesium alloys, both cast and wrought. The ease of obtaining the hardness values, the speed in operation, and the uniformity of results are all advantages in favor of the Rockwell machine.

The Rockwell-Brinell-Scleroscope relationships for magnesium alloys were obtained some time ago and the data published in an article, "Hardness Determinations on Magnesium Alloys," by Messrs. Grant and Winston in the May, 1930, issue of *Metals and Alloys*. The relation of Rockwell hardness "E" scale to Brinell hardness for magnesium alloys published in this article varies somewhat from the comparison for aluminum alloys presented by Mr. Templin. The corresponding values for a few of the Brinell hardness values are given below:

	ROCKWELL HARDNESS "E" SCALE	
BRINELL HARDNESS	MAGNESIUM ALLOYS	ALUMINUM ALLOYS
30	−16	11
40	34	41
50	59	59
60	72	70
75	84	82

In the lower hardness range, the Rockwell values for magnesium alloys are considerably lower than the Rockwell values for aluminum alloys for a given Brinell hardness. As the hardness increases, the difference in Rockwell readings for magnesium and aluminum alloys decreases until a Brinell hardness of 50 is reached. At this point the Rockwell readings are the same for both alloys. For Brinell hardness values above

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[1] Metallurgist, The Dow Chemical Co., Midland, Mich.

Geo. L. Smith

50, there is but little difference in the Rockwell values for magnesium and aluminum alloys.

MR. L. B. TUCKERMAN.[1]—All of this study emphasizes the fact that in the so-called hardness tests we are not measuring one property of the material. What is measured is dependent upon the shape of the indenting tool, upon the load, and upon other conditions of the test. The thing which we call hardness may be any one of a number of different combinations of more or less related properties. The fact that the relation between Rockwell and Brinell numbers for the magnesium alloys is different from the relation between Rockwell and Brinell numbers for the aluminum alloys—all these differences emphasize the fact that when we say hardness we should be very careful to qualify what we mean by hardness.

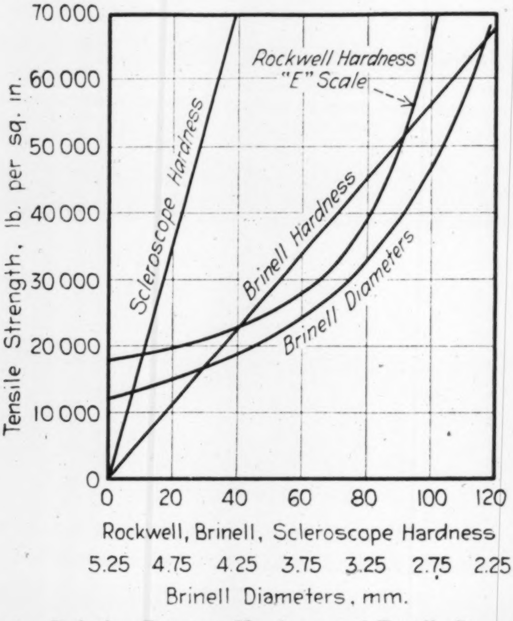

FIG. 1.—Relation Between Hardness and Tensile Strength.

MR. V. E. LYSAGHT[2] (*presented in written form*).—Tests made on the Rockwell tester reported in Mr. Templin's paper have been designated as "E" scale tests and "S" scale tests and he pointed out that the latter of these scales has not been recognized by the Society's Tentative Method of Rockwell Hardness Testing of Metallic Materials. The manufacturers of the Rockwell tester have now assigned values to all possible combinations of load and penetrator and at the next meeting of the Indentation Hardness Committee when the tentative methods for Rockwell hardness testing will be discussed, this matter will be taken up so as to prevent different investigators assigning different letters to the same combination of load and penetrator.

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[1] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.
[2] Wilson Mechanical Instrument Co., Inc., New York City.

In Mr. Templin's paper, it is pointed out that for light metals, the Rockwell tester, near the lower end of the scale, shows large variations in hardness values for a small change in tensile value and that at the upper end of this scale, the Rockwell hardness shows small variations for a large change in tensile strength. The curves showing the relation between the Brinell hardness number and tensile strength and scleroscope and tensile strength values do not show this to be so pronounced.

In the accompanying Fig. 1, the relation between the Brinell number and tensile strength is shown, which is the same as Fig. 5 of Mr. Templin's paper. If we now plot these same values, using Brinell diameters, the actual Brinell readable units (500-kg. load, 10-mm. ball), it will be seen that the curve takes the same form as the Rockwell-tensile curve. Since the Rockwell number is based on a linear depth measurement and the readable Brinell unit is based on a linear diameter measurement, we believe it only fair when comparing the two machines to compare them on the same basis. One cannot read Brinell 120, 119, 118, etc., but one can read Rockwell E 100, 99, 98, etc., the same as one reads Brinell diameters 2.30, 2.35, 2.40 mm.

It is our contention that specifications should be considered on actual readable units obtained on a hardness tester if hardness is to be used for a specification. By converting the readable units obtained and plotting them after they have been converted to a kilogram per square millimeter basis, a straight-line relation between the tensile strength and the hardness numbers may be obtained. Actually the material is being accepted on the readable units and by properly setting the specifications for the depth measurement (Rockwell numbers) or diameter measurement (Brinell numbers), the desired material may be selected.

The curve shown in Fig. 4 of the paper is the characteristic curve obtained when showing the relation between Rockwell hardness number and tensile strength and it is, in fact, the same type of curve that we obtain when showing the relation between Brinell and Rockwell numbers. The Bureau of Standards observed the same thing and in their publication "Relationships Between Rockwell and Brinell Numbers"[1] by S. N. Petrenko, showed the relation between the two tests by plotting the Rockwell hardness number against the reciprocal of the Brinell number.

In a previous investigation carried on by J. R. Townsend, W. A. Straw and C. H. Davis,[2] reported upon at the 1929 annual meeting of the Society, the same characteristic curves were obtained when comparing the Rockwell and tension tests for sheet non-ferrous metals, which included alloys of brass, nickel silver and phosphor bronze. When they observed that the Rockwell hardness - tensile strength curve became asymptotic so that there was little change in hardness for a large increase in tensile strength in the case of the harder tempers, then they increased the load used with that particular penetrator. Since this investigation several thousand tons of this sheet non-ferrous metal have been purchased on Rockwell specifications, and as nearly as we can ascertain, successful results have been obtained. We believe that in the present investigation, had the load been increased to 150 kg. after readings of approximately E 80 were reached, a better relation would have been obtained between the Rockwell hardness and the tensile strength.

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[1] S. N. Petrenko, "Relationship Between Rockwell and Brinell Numbers," National Bureau of Standards, *Journal of Research*, Vol. 5, No. 1, July, 1930, p. 19.

[2] J. R. Townsend, W. A. Straw and C. H. Davis, "Physical Properties and Methods of Test for Some Sheet Non-Ferrous Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 381 (1929).

Figure 7 of the paper is shown in the accompanying Fig. 1 when plotted to the same scale as the Rockwell test. In Table II, Mr. Templin has tabulated both the Rockwell hardness and the scleroscope hardness to the nearest half point and we believe that they should be plotted to the same scale for comparative purposes, and not to have one number on one curve represent twice the hardness number it does on another as in Figs. 4 and 7. Examination of our curve shows that the slope of the line obtained on the scleroscope is practically the same as on the "E" scale of the Rockwell test, even at its less sensitive part. Furthermore, we believe it to be generally accepted that the Rockwell tester can be read with more certainty to half a point than the scleroscope can be read to half a point.

In the accompanying Fig. 1, we have not shown the values taken on the "E" scale below the value of "E" zero because negative values should never be taken on the Rockwell tester as the instrument is not designed for such readings.

MR. R. L. KENYON[1] (*presented in written form*).—Mr. Templin has given us a clear idea of the factors involved in making hardness tests on aluminum and some of its alloys. No doubt some of these variables are peculiar to this material, but others are common to most metals, such as the effect of hard or soft surface layers.

The data presented on effect of thickness of specimen indicate a much lower limiting value for reliable tests than has been found for brass and ferrous metals.[2] In the absence of any explanation in regard to the method of selection and preparation of the samples reported in Table II, the reader naturally wonders what assurance there is that the specimens of each material are of uniform hardness and vary only in thickness. In the course of making the tests in the paper just referred to, it was found that the effect of thickness could be isolated only by reducing the thickness of a uniform piece of material by etching and polishing; cold rolling and annealing was found to result in specimens that varied in hardness as well as thickness. If the specimens reported in Table II were prepared by the latter method, it is possible that such variations may have been compensating, thus giving an unwarranted degree of confidence in the results on the thinner specimens. While it is possible that the cold working characteristics of aluminum may differ sufficiently from those of brass and ferrous materials to account for the much lower limiting thickness values for reliable hardness tests reported by Mr. Templin, it might be well to conduct a series of tests on uniform specimens prepared by the preferred method mentioned above before finally adopting the rather liberal thickness limits suggested by the author.

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[1] Research Metallurgist, The American Rolling Mill Co., Middletown, Ohio.

[2] R. L. Kenyon, "Effect of Thickness on the Accuracy of Rockwell Hardness Tests on Thin Sheets," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 229 (1934).

Attention is called to the data for material 2S-O presented in Table II. The Brinell and Vickers tests, which are based on diameter measurements after release of the load, show much less variation with thickness than the Rockwell results, which represent depth measurements under a minor (10-kg.) load. Is it not possible that the severe distortion on the thinner pieces, as shown in Fig. 1 of the paper, results in a diameter of impression measurement which is somewhat greater than it should be due to the curving of the specimen up around the ball? This would tend to offset the anvil effect.

The writer feels that some comment should also be made on Figs. 4 and 5. The inference is made that the Brinell hardness test is not subject to hypersensitivity with deep impressions nor insensitivity with shallow impressions. As a matter of fact, both the Brinell and Rockwell tests are made with spherical penetrators and both have these limitations in common. The only reason that the relationship shown in Fig. 5 is a straight line is that the Brinell hardness number is a derived value. If the diameter measurements (which are the original data obtained) are plotted against tensile strength, the relationship is similar to that shown in Fig. 4 for Rockwell hardness *versus* tensile strength. This emphasizes the necessity of using both tests, in such a way that neither hypersensitivity nor insensitivity of readings occurs. The manufacturers of the Rockwell machine have indicated these limits for the "B" scale for example to be about B-0 to B-100.

The difficulties with the Rockwell test mentioned on page 9 seem to apply with equal force to other indentation hardness tests and it seems, therefore, that the conclusion reached regarding the unsatisfactory nature of one particular test is hardly justified. Proper calibration is necessary for all such tests and it is just as important to use correct testing conditions with one as another.

MR. R. L. TEMPLIN.[1] Answering Mr. Kenyon's discussion, the specimens tested for the purpose of determining the effect of thickness on the hardness values obtained were of two types. In one type the specimens were prepared by machining thin specimens from thin plate specimens, always maintaining one original surface of the material on which the hardness values were taken. In the case of the other type of specimen, various thicknesses of material were used, starting with metal of the same composition and using a fabrication process which would give exceptionally uniform tensile properties in the various thicknesses used as specimens.

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

I do not believe it is probable that the cold working characteristics of aluminum are sufficiently different from those of brass and the ferrous metals to account for the much lower limiting thickness values found. On the other hand the coefficient of friction existing between the aluminum alloys and steel is appreciably higher than in the case of brass and perhaps in the case of steel against steel with the result that there is a lateral restraining force due to the higher coefficient of friction which would in effect permit of thinner specimens when testing aluminum. It is also quite probable that severe distortion on the thinner pieces, as shown in Fig. 1, results in a diameter of impression measurement which is somewhat greater than it should be due to the curving of the specimen up around the ball as suggested by Mr. Kenyon.

His comments on the necessity for using both the Rockwell and the Brinell test in such a way that neither hypersensitivity nor insensitivity of a reading occur is quite in agreement with the author's ideas.

The points brought out by Mr. V. E. Lysaght are very helpful. It is quite true that changing the ball and load combinations on the Rockwell machine will very much improve the character of the hardness values obtained. The use of so many different combinations, however, is, in the author's opinion, one of the objections to the use of the test because of the possibility of confusion or error. The negative hardness values were included in the data given because they can be obtained with the machine and because they are sometimes met with in the commercial field.

A NEW METHOD AND MACHINE FOR DYNAMIC HARDNESS TESTING

By W. M. Patterson[1]

Synopsis

This paper points out the need for a reliable hardness scale with definite physical units of equal value throughout the scale. It comments briefly on previous work in this field showing reasons for the inadequacy of past methods.

A new machine for dynamic hardness testing is described and the development of a scale fulfilling the desired conditions is explained.

Test data are given on alloys of lead, copper, and aluminum, cast iron, and soft and medium-hard steels. The reproducibility of results to be expected with the method in laboratory and production work is estimated, and a brief discussion of the advantages and particular field of use of the machine is given.

Introduction

For many years there has been evidence of a desire in the commercial field for a method of hardness testing that will show a correct correlation between results found in testing soft materials and results obtained in testing hard materials. The ideal comparison can best be presented by the use of a hardness scale in which actual physical values are used rather than an empirical scale. The use of a gradually applied load presents many difficulties in reducing the work done on the test specimen to terms of expended energy, so in attempting to develop a definite scale, a dynamic method of test has been used.

The use of dynamic methods of hardness testing is of course not new. Mr. W. I. Ballentine did some research with dynamic testing in the early nineteen hundreds, and received the John Scott Medal, on recommendation of the Franklin Institute in 1908, for his work in development of a dynamic hardness tester at that time. Considerable interest in this work was shown in England and Europe in the period from 1915 to 1920.

Professor C. A. Edwards[2] of the University of Manchester was one of those most actively interested at that time, and the author owes much to his findings published on this subject. Edwards' original conclusion was that the diameter of indent was equal to a constant (dependent on the metal and indicative of its hardness) times the fourth root of the energy causing the indent, but later concluded that an additional correction was needed in order to truly represent hardness. About this time Col. M. Martel[1] concluded that for a given hardness, the volume of indent was proportional to the energy of impact independent of the shape of the penetrator, and that the ratio of impact energy to volume of indent was a constant for a given material, this constant being an indication of the hardness. Edwards carried on experiments along this line using 5, 7, 10 and 15-mm. balls, and cones of approximately 90, 53, 37, and 28 deg. included angle. A series of experiments on different metals showed that the laws given by Martel did not hold exactly in experiment, for it was found that, with a given indenter on a given metal, the ratio of energy to volume decreased as the energy was increased, and that with a given energy of impact and a given material the ratio of energy to volume decreased as the diameter of the indenter decreased.

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[1] Research Engineer, Tinius Olsen Testing Machine Co., Philadelphia, Pa.

[2] C. A. Edwards, "The Resistance of Metals to Penetration Under Impact," *Journal*, Inst. of Metals, Vol. XX, p. 61 (1918).

C. A. Edwards and F. W. Willis, "A Law Governing the Resistance of Metals when Tested by Impact with a 10-mm. Steel Ball, and a New Hardness Scale in Energy Units," *Proceedings*, Inst. Mechanical Engrs., Part I, p. 335 (1918).

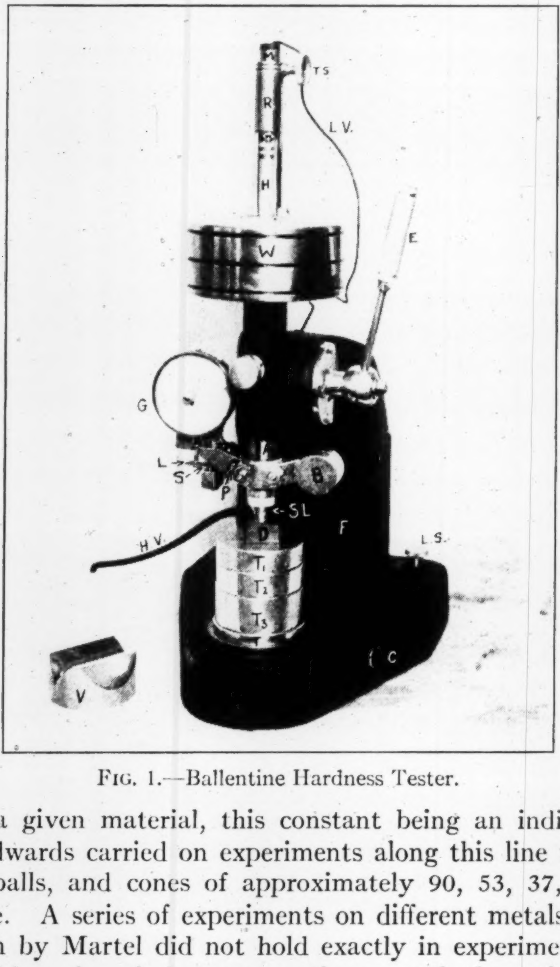

FIG. 1.—Ballentine Hardness Tester.

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[1] M. Martel, Commission des Methods d'Essai des Materiaux de Construction, Tome iii, Section A ("Metaux"), p. 261 (1895).

In this work all attempts to reach a hardness scale have evidently been attempted by employing a single equation, and in the opinion of the author, this was the cause of the failure to obtain satisfactory results, for both the resilient energy and the resistance to permanent deformation must be taken into account, and these two factors do not depend on the same physical characteristics of the indenter.

DESCRIPTION OF THE BALLENTINE HARDNESS TESTER

The hardness tester used in the present investigation was invented by Mr. W. I. Ballentine and developed by him over a period of years. The instrument as built by the Tinius Olsen Testing Machine Co. is shown in Fig. 1.

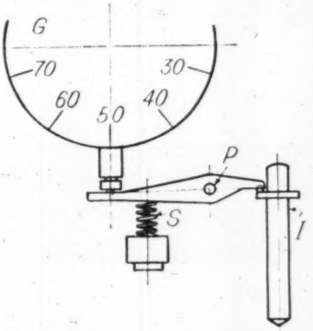

FIG. 2.—Diagrammatic Sketch of Ballentine Recording Mechanism.

Referring to the figure, *F* is a heavy cast iron frame carrying a working table *T*, which is adjusted until horizontal by means of two leveling screws *L.S.* The table is in a plane at right angles to the axis of a very accurately drilled rifle tube *R*. This rifle tube may be moved up or down by means of a rack and pinion actuated by lever *E*.

A diamond point indenter, *I*, is held centered on the axis of the rifle tube by the sleeve *SL*. This indenter is held in constant contact with the test specimen by its own weight plus the action of the spring *S* on the lever *L* which is pinned at *P*. A diagrammatic sketch of this part of the machine, which is hidden by the sleeve and gage mechanism, is shown in Fig. 2.

Any movement of the indenter is followed by the lever *L* and is registered by the action of the lever on the gage *G*. The weight of the gage mechanism is balanced by the counter-weight *B*. A magnet *M*, adjustable by means of thumb screw *T.S.* to give any height of hammer drop between 0 and 70 mm. allows the hammer *H* to be released for a free fall to strike the indenter. This is accomplished by breaking the circuit at contact button C. The height of fall of the 320-g. hammer is fixed to give the desired energy of impact by means of gage blocks inserted in slot A in such a manner that the gage block is in contact with the top of the indenter and the base of the hammer. Specimens of different heights are provided for by the addition or removal of blocks T_1, T_2, and T_3. For testing specimens having no flat base the V-shaped holder V is used. Current at 110 v. enters through a high-voltage lead and is reduced in a rectifier to low voltage before going to the magnet.

THEORY OF THE BALLENTINE HARDNESS SCALE

Since hardness is an inherent property of a material, if the condition of the material is unchanged, it should make no difference what the conditions of test, such as the form of the indenting tool or the energy of impact, the same hardness value should result. Referring to the Ballentine hardness tester, let us consider how the energy of the indenter at impact is dissipated. Part of this energy is expended in a permanent deformation of the test specimen, an easily measured geometric property. It is logical that the permanent deformation should bear a direct relation to the energy absorbed in causing that deformation.

All the energy, however, is not absorbed, part being rejected as rebound, or dissipated as heat of friction, and possibly other forms of lost energy. In the opinion of the author, as based on observation from experiment, the rebound energy factor is the only one of these factors large enough to be of importance in reproduction of results in this type of test.

In analyzing the energy used up in rebound let us start with the fundamental equation:

$$F = \frac{W}{g}\,\frac{V_2 - V_1}{t} \quad \ldots\ldots\ldots\ldots (1)$$

where F = average vertical component of force exerted during elastic recovery,
W = weight of the hammer,
g = the acceleration of gravity,
V_2 = the velocity of the hammer on leaving the specimen,
V_1 = the velocity at the start of the rebound, and
t = the time of elastic recovery.

In addition let
E_r = the energy of rebound,
f = average vertical component of force per unit projected area,
A = projected area of the indent,
I = depth of indentation, and
C = a constant dependent on the cone angle.

V_1 is zero, so we may say:

$$F = \frac{W}{g}\,\frac{V_2}{t} \tag{2}$$

Transposing and dividing by 2:

$$\frac{W}{2g} = \frac{Ft}{2V_2} \tag{3}$$

The energy of a similar weight falling the height of rebound:

$$E_r = \frac{W}{2g}\,V_2^2 \tag{4}$$

By substitution from Eq. 3:

$$E_r = \frac{Ft}{2V_2}\,V_2^2 \text{ or } E_r = \tfrac{1}{2}\,FtV_2 \tag{5}$$

$$F = fA \tag{6}$$

This may be expressed for a conical indentor as:

$$F = 2\,CfI^2 \tag{7}$$

By substitution of Eq. 7 in Eq. 5, we obtain:

$$E_r = CfI^2V_2t \tag{8}$$

Equation 8 shows that the rebound energy will depend on the form of the indentor, the projected area of the indent, the average force exerted on the indenting surface, and the velocity and time of recovery of the specimen.

Figures 3 (*a*) and (*b*) show the effect of the form of the indenter. It is seen that the force normal to the surface at any point on the indenter may be resolved into a horizontal force and a force tending to throw the indenter back up. Since a cone must give similar triangles of force it is mathematically a more convenient form of indenter to use than a ball, for C in Eq. 8 would not be a constant for a ball indenter, as well as difficulties brought in by changing angles of force.

From the preceding discussion we conclude that the consumption of energy can be split up into that portion measured by the volume of permanent deformation, and that rejected, as measured by a rebound energy factor dependent on the material and the projected area of deformation, provided the resolution of force angle does not change as the indention progresses.

Presented in the form of an equation we have:

$$H = \frac{E_t - I^2\,R}{V} \tag{9}$$

where H = the Ballentine hardness number in inch-pounds per 0.001 cu. in.,
E_t = the total energy of impact in inch-pounds,
I = the depth of deformation in 0.1 in.,
R = a resiliency factor inherent in the material, expressed as unit force × time of recovery × speed of recovery, and
V = the volume of deformation in 0.001 cu. in.

It is seen from this equation that the hardness of the material is defined as the energy causing a deformation divided by the volume of that deformation and that the resilience of the metal does not enter into the definition. The resiliency factor varies with the energy causing the deformation and also varies with different forms of indenter and accordingly should be eliminated in a determination of hardness. This elimination of the resiliency factor in testing is important, for the variability of its effect in the resistance of metals to indentation in commercial use, where energy of impact and the form through which the load is applied vary widely, makes results with a specialized condition misleading.

TEST PROCEDURE

The Ballentine hardness tester affords a quickly and easily operated instrument. Referring again to Fig. 1, the test specimen is placed on the anvil and the indenter lowered until it is in contact with the specimen. The hammer is then adjusted for the desired height of fall, released, and the depth of penetration read on the gage which is graduated to read the nearest 0.00005 in.

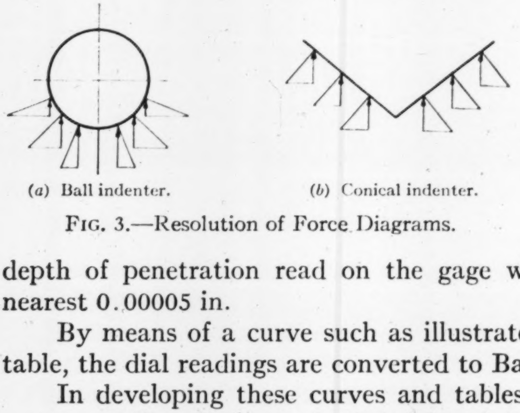
(a) Ball indenter. (b) Conical indenter.

FIG. 3.—Resolution of Force Diagrams.

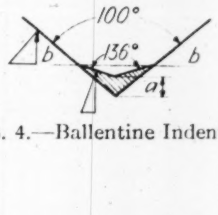

FIG. 4.—Ballentine Indenter.

By means of a curve such as illustrated in Fig. 6, or of a conversion table, the dial readings are converted to Ballentine hardness numbers.

In developing these curves and tables, tests were made in the usual way, recording, however, both height of drop and depth of penetration or dial reading. Between five and ten thousand readings were taken on about forty different test specimens. The final valuations set forth in the curves and tables in this paper represent ten readings from each of four heights of drop, made on four lead alloy specimens, two copper alloy specimens, four aluminum alloy specimens, one cast iron specimen, and six steel specimens, a total of 680 tests on 17 samples covering the range from the very soft to the medium hard.

With the data available, an attempt was made to find how R in Eq. 9 varied for different metals. Since hardness has been defined as a property of the material independent of the method of test, attempts were first made to reach a consistent relationship between absorbed and unabsorbed energy by substituting in Eq. 9 known quantities for E_t, I, and V. This procedure gave a fairly smooth curve when values of R were plotted against E_t/V for soft materials, but lack of uniformity of the hardness of the materials caused very marked variation in the case of hard metals. However, it was found that values of $I^2 R$, for soft metals, as obtained from the average curve, developed as explained above, were nearly a constant of 5.55 in-lb. for a 10-mm. drop.

TABLE I.—ILLUSTRATING THE DEGREE OF UNIFORMITY OF BALLENTINE NUMBERS WITH DIFFERENT ENERGIES OF IMPACT.

Material	Drop, mm.	Ballentine Hardness Number[a]	Spread, per cent
Lead	10	19.9	
	20	19.8	
	25	19.3	
	30	19.4	2.6
Babbitt	10	68	
	20	69	
	25	67	
	35	66	4.4
Babbitt	10	91	
	20	91	
	25	89	
	35	88	3.3
Aluminum	10	128	
	20	128	
	30	126	
	70	123	4.0
Babbitt	10	145	
	25	146	
	30	149	2.7
Copper alloy	10	182	
	20	183	
	25	183	
	50	183	0.6
Aluminum alloy	10	264	
	20	261	
	35	256	
	70	253	4.3
Aluminum alloy	10	268	
	20	269	
	35	269	
	70	269	0.4
Aluminum alloy	25	386	
	35	378	
	50	381	
	70	378	2.1
Cast iron	25	452	
	35	450	
	50	455	
	70	441	3.1
Steel	25	510	
	35	510	
	50	494	
	70	490	4.0
Steel	25	652	
	35	623	
	50	629	
	70	650	4.5
Copper alloy	25	799	
	35	767	
	50	768	
	70	773	4.0
Steel	25	878	
	35	859	
	50	845	
	70	840	4.4
Steel	25	980	
	35	975	
	50	978	
	70	970	1.0
Steel	25	997	
	35	1010	
	50	1010	
	70	980	2.9
Steel	25	1012	
	35	987	
	50	1002	
	70	1008	2.4

[a] Each Ballentine number is computed from the average indentation of ten tests from the fall listed for that number.

Assuming this to be the correct value and using it to solve for hardness, and then equating for other heights of drop, it was found that an almost constant value of unabsorbed energy for each height of drop resulted and that the equation of the curve of this energy factor plotted against height of drop was very close to the parabolic function:

$$(\text{unabsorbed energy})^2 = 2\ P \times \text{height of drop.}$$

However, when these values were used in plotting R for different heights of drop against E_t/V, a series of curves resulted lying close together but with the curves for high falls lying slightly below those for low falls. An average was taken of this series, giving the curve shown in Fig. 5. Values of R taken from this curve and used in Eq. 9 gave a closer agreement of results with different impact energies than any method tried previously.

ILLUSTRATIONS FROM ACTUAL TEST

Figure 4 illustrates the diamond indenter used in the actual test. As will be seen, a small portion near the point is cut at a 136-deg. angle to give the indenter higher shock resistance. As computations are based on a 100-deg. cone, an original setting is made to compensate for distance *a*, and factors are included in the computations to correct for variations due to the cross-hatched area and the difference in angle of face on which the rebound force acts up to cross-section *b-b*.

Figure 5 is the curve representing values of R for different values of total energy divided by volume. Below is a sample computation and illustration of results for an aluminum alloy sample having a Brinell hardness of eighty with a 500-kg. load. The same terminology as used in Eq. 9 applies and in addition E_a represents the absorbed energy in inch-pounds, and E_r represents the unabsorbed energy in inch-pounds.

Drop = 10 mm. = 0.3937 in.
Dial Reading = 0.0846×10^{-1} in. indentation.
$I^2 + 0.004$ (136-deg. angle factor) = 0.0074×10^{-2} sq. in.
$V = 1.5\ I^3 - 0.00006$ (136-deg. angle factor) = $1.5 \times 0.00067 - 0.00006 = 0.00085 \times 10^{-3}$ cu. in.
Weight of hammer = 320 g.
$E_t = 0.320 \times 2.2046 \times 0.03937 \times 10 = 0.278$ in. lb.
$E_t/V = 0.278 \div 0.00085 = 327$ in-lb. per 0.001 cu. in.
R from curve = 6.72 in-lb. per 0.01 sq. in.
$I^2 \times R = 0.0074 \times 6.72 = 0.050 = E_r$ in-lb.
$E_a = E_t - E_r = 0.278 - 0.050 = 0.228$ in-lb.
Ballentine hardness number, $E_a \div V = 0.228 \div 0.00085 = 268$ in-lb. per 0.001 cu. in.

Drop, mm.	I, in. $\times 10^{-1}$	$I^2 + 0.004$, sq.in.$\times 10^{-2}$	V, $1.5\ I^3 - 0.00006$, cu. in. $\times 10^{-3}$	E_t, in-lb.	E_t/V, in-lb. per cu. in $\times 10^{-3}$	R, Curve Values	E_r, $I^2 \times R$, in-lb.	E_a, $E_t - E_r$ in-lb.	Ballentine Hardness Number, in-lb. per cu. in. $\times 10^{-3}$	Spread
10	0.0846	0.0074	0.00085	0.278	327	6.72	0.050	0.228	268	269-268 or 0.4 per cent
20	0.1068	0.0118	0.00178	0.556	310	6.52	0.077	0.479	269	
35	0.1296	0.0172	0.00320	0.972	304	6.38	0.110	0.862	269	
70	0.1645	0.0275	0.00660	1.946	295	6.21	0.171	1.775	269	

Table I gives results found on a series of metals ranging from soft lead to medium hard steel. The average percentage spread is 2.98 per cent, which is well within the hardness variation of the metals used. By use of Fig. 5, curves of Ballentine hardness number *versus* indentation for different heights of drop can be obtained. Figure 6 illustrates these curves.

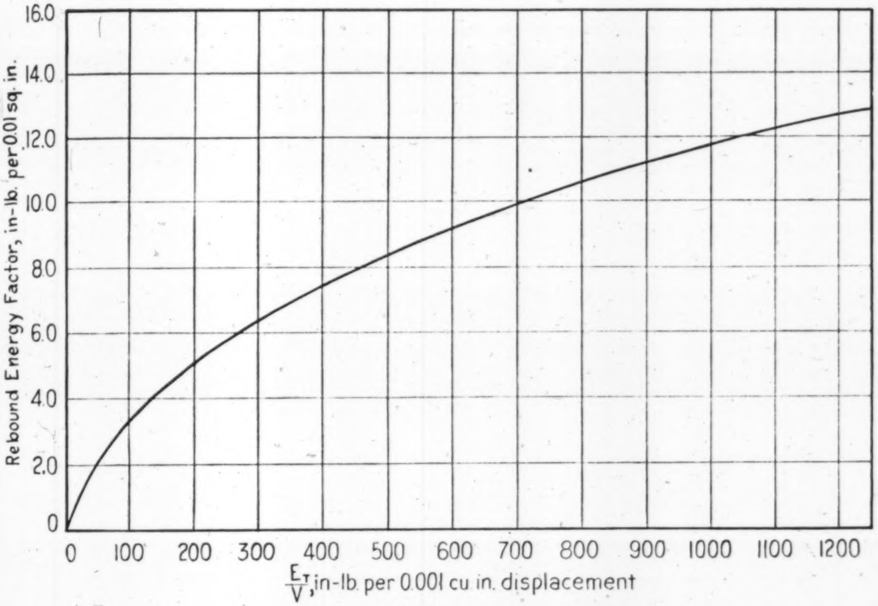

FIG. 5.—Curve of Rebound Energy Factor.

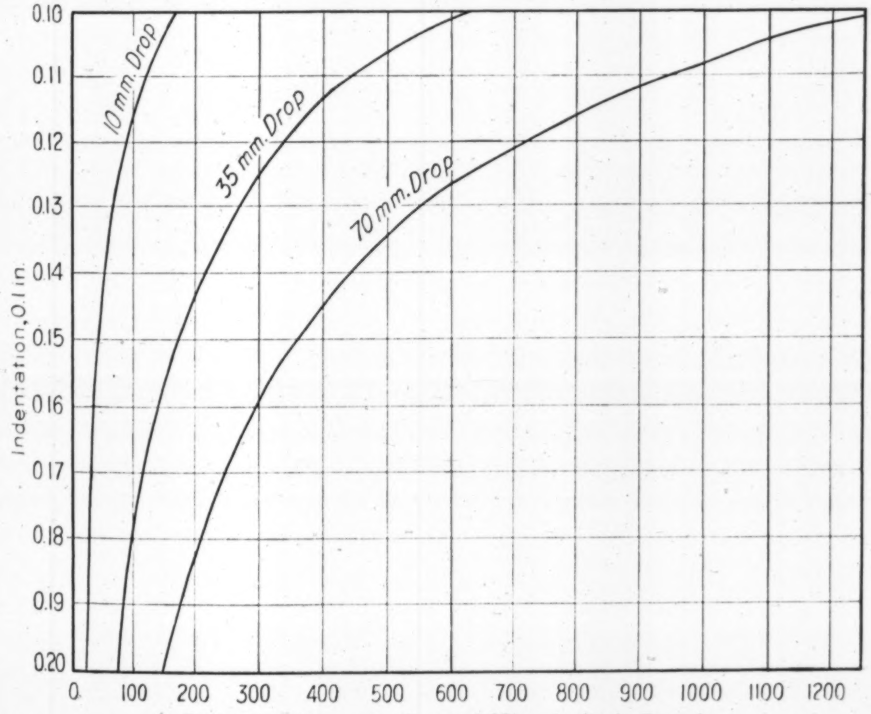

FIG. 6.—Relation of Ballentine Numbers to Depth of Indentation.

ACCURACY OF THE BALLENTINE SCALE

Table I shows a trend of slightly decreasing values for E_a/V as the energy of impact increases. This indicates that as indention progresses, the resilient energy decreases slightly from a direct proportionality to diameter of the indentation.

The accuracy to be expected in laboratory testing as shown by the average spread of hardness values is 2.98 per cent. If the average of the four values found for each material is considered to be the correct hardness, then the variation shown from these averages will be of the order of one-half the spread. When the machine is used as a laboratory instrument, by taking ten or more readings an accuracy within 3 per cent can be expected on any material within the range of the machine. Here accuracy has been used in the sense of comparison with the expected average hardness of the test specimen and therefore takes into account lack of uniformity of the test specimen as well as possible discrepancies of the machine. The possible magnitude of this discrepancy seems very slight as the form of the indenter, and the weight of hammer are constants, and the gage blocks are quite accurate. The accurately drilled rifle tube seems to permit a free fall so it is concluded that variations from the average represent, in the main, variations in hardness from point to point on the specimens tested. On specimens of markedly uneven hardness, less consistent results will be obtained, while on uniformly hard specimens a higher percentage of accuracy may be expected. Soft materials appear to be more uniform in hardness than the harder materials, which is responsible for higher accuracy on soft materials.

Some idea of the probable accuracy to be expected in commercial testing, where only one test on each specimen is made, can be gained by noting the maximum variation in the ten readings. This shows that even with not very uniform hardness of the specimens, reproducibility within 6 per cent can be expected.

SOME ADVANTAGES OF THE BALLENTINE SCALE AND METHOD

The method of testing described affords a hardness scale with definite physical units of measurement, inch-pounds absorbed in causing a permanent deformation of 0.001 cu. in. This means a hardness scale in which each unit has the same valuation as any other unit on the scale. The scale has been developed to be substantially independent of the height of drop, making the uniformity of scale hold throughout so that hardness numbers of all materials tested are open to direct comparison even though tested with different heights of drop and at the extremes of the testing range of the machine. Since the time of load application is practically eliminated as a factor, the field of comparison of hardness of metals at high temperatures where the metals are in a highly plastic state is opened. The tester works with almost equal accuracy from the softest commercial metals up to hard steels. The speed of making tests is many times faster than any of the commercially used static tests. In production testing, a pair of adjustable tolerance indicators may be employed which makes possible the setting of limits in checking the acceptibility of a run of material without reference to the hardness scale for each reading, thus speeding up the test still further. In production testing, heights of fall are taken to give depths of indentations between 0.01 and 0.02 in., which small impression would mar the work only slightly.

For the machine used, a scale has been developed which is substantially independent of the height of drop. However, the author feels that only time and further experiment can test the value of the theory for all cases of resistance to penetration under impact. He is confident, however, that the present work represents a step in the right direction, and that it has proved that in hardness testing, absorbed and rebound energy must be treated separately.

DISCUSSION

MR. R. L. KENYON[1] (*presented in written form*).—Although there are already many different hardness tests, the perennial suggestions for additional ones indicate that the last word in hardness testing is yet to be said. Continued searching for new and better tests should be encouraged, but new methods should not be adopted in material specifications until the various factors influencing the tests are well understood.

One stumbling block in this field is the lack of agreement on a definition of hardness. Some definitions express the hardness of a material as its resistance to deformation (indentation) or to abrasion. The author proposes to define it as the energy consumed in making an indentation with a standard penetrator. In either case it is well to remember that the resistance to be measured (or the energy consumed in overcoming this resistance) is that which the material exerts against a complex distribution of stresses at the point of application of the deforming tool. The numerical value determined will depend on the material and on the distribution of the stresses under and around the penetrator. For this reason it is believed that the only solution is to set up experimental conditions such that a similar state of stress is imposed on the specimen every time a hardness test is made and to determine the behavior under this standard condition. In other words, the hardness test probably will always be an arbitrary test.

The author refers repeatedly to his definition of hardness as "a property of the material independent of the method of test" and to the ability of his test to measure an "inherent" property of the material. This seems to be a case of the wish being father to the thought because there is no reason to believe that the energy consumed in making an indentation (grant that it could be determined) would be independent of everything except the material. The size and shape of the penetrator and the speed of loading would all have to be investigated before any such broad statement could be accepted. Furthermore, hardness is not any single property, but depends on elasticity, plasticity, and rate of work hardening.

In addition to these general comments on the author's proposal for a test which measures the "inherent" hardness of a material, there are several specific statements in the paper which warrant further discussion. The reasoning is not at all clear upon which the author bases the statement that the permanent deformation (meaning the actual indentation) should bear a direct relation to the energy absorbed in causing that deformation. The energy is utilized not only in producing in the surface a depression of definite shape and measurable dimensions, but also in moving the metal under and around this depression. The actual volume of metal moved is much larger than the depression itself and depends on the plastic flow and work hardening characteristics of the particular material under test. The resistance to movement offered by the metal under and around the depression will also vary according to these characteristics. There is no apparent reason for believing that there should be a definite relationship between the volume of the depression and the energy expended in deforming all of the metal under and around the impression.

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[1] Research Metallurgist, The American Rolling Mill Co., Middletown, Ohio.

In the discussion on page 309 reference is made to Fig. 3, which shows the resolution of forces at the interface between the specimen and the penetrator. For the conditions shown in Fig. 3 and assumed in the discussion, it is necessary that the friction between penetrator and specimen be zero. This is not true in actual testing and the assumption made by the author seems unjustified.

The method of eliminating the resiliency factor is not apparent. It is stated to vary with the energy expended in making the impression—but this is the unknown quantity. In other words, the author has one equation in two variable unknown quantities. It is possible that the rebound energy might be better evaluated by direct measurement. This might be accomplished by determining the height of rebound.

In discussing the advantages of his proposed test, the author gives no comparisons between Ballentine Hardness values and any of the accepted hardness scales which makes it difficult to appraise his statement that it gives a uniform scale in which "each unit has the same valuation as any other unit on the scale." This much can be said for the Brinell or Vickers test in which the load is divided by the area of the impression.

MR. S. R. WILLIAMS[1] (*by letter*).—The paper is in the right direction for the improvement of indentation instruments. Such studies should be encouraged and extended, because indentation methods are so widely used at present. If hardness is to be defined as resistance to penetration, the method under discussion is very much to the point.

The writer, however, is one of those who believes that the time has come for some fundamental research on the problem of hardness, if hardness is to be used as the criterion whereby materials may be judged as to suitability for use.

For example, how hard is a snow drift? At once the answer is at hand. It is the resistance offered to the penetration of a snow plow. This resistance to penetration, however, will depend upon the angle between the two wings of the snow plow. A 30-deg. angle will penetrate more easily than a 60-deg. angle. Hardness, therefore, becomes a function of the snow plow as well as of the material tested, whether a dynamic or a static method is employed in driving the snow plow into the drift.

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[1] Professor of Physics, Fayerweather Laboratory of Physics, Amherst College, Amherst, Mass.

Carrying on this illustration of the snow plow and the drift, what are the factors which produce resistance to penetration? This may be answered by an equation: HARDNESS = some function of the penetrating point + that function which depends upon tearing the crystals of snow apart (brittleness) + some function of friction between the crystals of snow sliding over each other (ductility), + some function of the friction of the crystals sliding over the surface of the wings of the snow plow.

$$H = f(\phi) + f\left(\frac{m_1 m_2}{r^2}\right) + f(\mu_1 p) + f(\mu_2 p)$$

Just one more example from the snow drift will indicate how far the penetration method is from being based on something fundamental and basic.

The fourth factor indicates that a rusty snow plow will have greater resistance offered to its penetration than a polished one. Once more hardness is a function of the penetrator rather than of the penetratee. This same factor is observed in any ball or cone indenter. Measure the hardness of a steel surface by taking readings over an extended surface and then repeat with oil on the surface. The hardness will be increased by the oil to as much as 4 per cent in some cases. The oil allows the steel to flow around the ball rather than crack and break as when dry. The hardness as measured against plasticity is always greater than against brittleness. The Indian in making his arrowheads demonstrated this fact long ago. By means of his bone tool he could shear off flakes of flint from the main piece, where, by no manner of means could he cause the bone to penetrate the flint.

Modern physics has advanced to a point now where it seems to the writer that the basic factors of hardness should be emphasized. It is only as we combine modern science with metrology may we hope to advance the art of measuring hardness.

MR. ERICH FETZ[1] (*by letter*).—For those of us who have actually done some work on the development of an impact hardness tester, Mr. Patterson's paper is of particular interest. The author discusses the need for a "reliable hardness scale with definite physical units of equal value throughout the scale" and comments briefly on previous work in this field showing reasons for the inadequacy of past methods. Unfortunately, reference is only made to the results of Martel and Edwards[2] available in the English language, whereas the partly fundamental investigations of Wüst and Bardenheuer, Sauerwald and co-workers, Körber and Simonsen, Schneider, Düsing, Franke, Ehmke, Roos, Brinell and Dillner, Gessner, Liepe, Örtel and Pölzguter and others are not mentioned.

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[1] Research Metallurgist, Wilbur B. Driver Co., Newark, N. J.

[2] Edwards also uses a hardness scale in energy units in which each unit or any portion of the scale has the same significance as those on any other section.

The utilization of the drop hardness testing method in German research laboratories had a very practical aim, namely, to develop a simple method for testing the strength of metallic materials at elevated temperatures. At that time, the difficulties introduced by the use of steel balls in the static test were not overcome. Naturally much attention was paid to the interrelation between the new dynamic and the standard static hardness testing methods, to the source of errors in the dynamic testing machine and to the significance of the energy lost in the rebound of the drop weight. The marked difference between Mr. Patterson's testing machine and most of the European dynamic hardness testers is that the latter apply much greater impact energies. The principal reasons for the European trend were to overcome the difficulties involved in determining microscopically the diameter of the indentation, to utilize a larger indenting tool less subject to deformation, to minimize the effect of structural inhomogeneities of the material under test, and last but not least to increase the permanently deformed region of the testing material at the expense of the elastically deformed region, or in other words to reduce the interfering effect of the rebound with respect to different kinds of material. It was the object of the dynamic hardness tester to determine the resistance to penetration or the "specific displacement work" and not to determine the elastic properties which enter the picture to a more and more pronounced degree the smaller the indentations.

The extreme case of a dynamic hardness machine with "negligibly" small indentations is the type of test based on the rather wide assumption that the rebound is the higher the harder the material under test. Mr. Patterson's new machine stands between this type of test and the European dynamic hardness apparatus. It has been established that hardness values obtained with the type of test based on rebound only furnishes the poorest relationship to other hardness values for the reason that the elastic properties exert too great an influence upon the testing result. With a great variety of metallic materials results comparable to dynamic and static hardness values may, however, be secured if the energy lost in the permanent deformation is taken into account or is held at a constant value. The energy lost in the minute impression varies considerably according to the material under test, and this leads us directly to the inherent shortcoming of Mr. Patterson's dynamic hardness testing machine, which makes the reverse mistake, namely, of inadequately accounting for the energy lost in the rebound of the drop weight. A calibration curve shown in Fig. 5, which admittedly represents an average taken on a series of curves, is used to "eliminate" the resiliency factor. The writer contends that the most accurate dynamic hardness testing method determines for each individual material and for each individual test the permanent deformation and the energy lost in the rebound.

It is significant that the rebound energy portion not only varies with the height of fall and the material under test, but also with the same material depending on structure and surface conditions. This discrepancy becomes even more pronounced at elevated temperatures as the exhaustive tests of Sauerwald and his school established definitely. The suggestion of Mr. Patterson to utilize his testing machine for high temperature work is not feasible with the present "curve of rebound energy factor" which only deals with rebound energy losses *summarily*. The determination of the actual working energy

$$\text{weight} \times (\text{height of fall} - \text{height of rebound})$$

would furnish a more nearly correct hardness value. The additional determination of the rebound height for each individual measurement would complicate the testing machine, it is true, but it would greatly widen its scope of utilization, and improve its accuracy and value as a physical testing instrument. Mr. Patterson states that attempts were first made to reach a consistent relationship between absorbed and unabsorbed energy but that "lack of uniformity of the hardness of the materials caused very marked variation in the case of hard metals." The same observations and conclusions had been arrived at by the writer. However, a simultaneous hardness determination with the Rockwell or Vickers machine disclosed that it was not a pronounced "lack of uniformity of hardness" but the deficiency of the dynamic hardness tester which was responsible for inconsistent results. Regarding the sources of errors in impact hardness machines, the energy consumption is not restricted to deformation and rebound, but further losses are due to vibration of the testing apparatus and sample, friction of the indenting tool which does not fall freely or is being rejected under an angle, stress propagation through the testing machine to "earth," heat losses at the surface of the sample and others.

The vibration of the testing machine is quite serious and is partly responsible for the fact that there is often not a perfect uniformity of readings on different machines, necessitating an individual calibration. In this connection, it would be interesting to learn whether identical readings are obtained on Mr. Patterson's machine when it is resting on its entire frame or on the leveling screws. It would seem that the small leveling screws should be liable to deform, to make indents on their resting platform or cause the whole machine to shift under heavier impacts. According to the writer's experience, a heavy, exceedingly rigid machine with special devices for clamping the specimen is necessary for reproducible results. Naturally energy is lost if the test specimen is allowed to bounce and in some dynamic testing machines using heavier impact energies samples are thrown clear from the anvil.

In the usual impact testing machines, an apparently important source of energy loss is the varying loss through transference to "earth" due to propagation of stress through the testing machine. The time of contact or striking velocity (0.001 to 0.004 sec. for the Charpy machine) appears to be quite important in view of the fact that the two principal velocities at which stress is propagated through steel are of the order of 17,000 and 10,500 ft. per sec. In the case of the dynamic hardness tester, where the direction of the indenter is completely reversed, the time of contact is longer. It appears that investigations along these lines may yield some important clues as to the source of energy losses in dynamic hardness testers. In this connection, the writer feels that some tests are also necessary to determine the effect of the elastic properties of the indenting tool, in other words, to compare hardness values secured with steel and diamond cones. The conclusions of Ludwig regarding the use of a 90-deg. cone in lieu of a steel ball for the static hardness test probably hold also for the dynamic testing method. It is not a happy step from a scientific point of view to change the diamond point so that geometrically similar indentations are no longer obtained. Mr. Patterson indicates that a small portion near the point of his 100-deg. cone was cut at a 136-deg. angle to give the indenter higher shock resistance. It would be interesting to learn how the diamond cone as a known brittle material stood up in those tests carried out with maximum drop heights and hardest steels.

It is very desirable that a scale of hardness should be rational, although it is true that non-rationality does not prevent a hardness scale from being very useful.

A critical discussion necessarily dwells upon the shortcomings of a new invention. Rather than point out all the various points on which perfect agreement exists, the writer wishes to congratulate Mr. Patterson on his highly interesting paper. Mr. Patterson's bold attack on the dynamic hardness problem certainly contributed more to a final solution of this kind of physical testing method than those people who are just sitting and watching on the side-lines.

MR. W. M. PATTERSON.[1]—The author wishes to thank Messrs. Kenyon, Williams, and Fetz for the discussions which they have presented. Each discussion indicates new fields of experiment in dynamic hardness testing.

Mr. Kenyon has brought up the matter of hardness (meaning the resistance to penetration) depending on more than one quality of the material, and the author is in agreement with him. Mr. Kenyon lists plasticity, elasticity, and work hardening as three factors. All of these have been considered in the presentation, plasticity as that factor determined by the Ballentine tester, elasticity as that factor found essentially constant for all metals tested for a specific impact energy, and work hardening a factor in repeated drops but not in evidence in single drop tests, due to the small impression and speed with which it is made.

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[1] Research Engineer, Tinius Olsen Testing Machine Co., Philadelphia, Pa.

Mr. Kenyon states that with the Vickers test and the Brinell test, using load divided by spherical area, a uniform scale exists. However, with most materials, changing of the load, or of the time the load is applied, will make this condition very hard to get. That a uniform scale exists for the Ballentine machine is evidenced by the consistency of results as illustrated by Table I.

It might be mentioned that the trend of comparison between the Ballentine and Brinell scale is a straight line, that experiments with different lubricants showed no difference in results, that the resiliency factor was found for the Ballentine indentor to be proportional to the applied energy, a known quantity, and that the energy *versus* deformation relation has been found, in all experiments made by the author and by Mr. Edwards, regardless of form of indentor and amount of applied energy, to be very nearly proportional even without a separation of plastic and elastic properties.

In Mr. Williams' discussion the energy to displace a given amount of snow rather than the force to drive different angle plows should be the consideration. The other points raised by Mr. Williams are covered in the discussion of Mr. Kenyon's comments.

In discussion of several points raised by Mr. Fetz, the author feels that the size of the indentation in comparison to the applied energy is the important consideration, rather than the amount of energy alone, in determining the relative importance of plastic and elastic effect. Due to the magnetic release construction, the hammer falls freely, and with the constant contact of the actual indentor angularity at impact will not occur. The heavy weight, *W*, Fig. 1, acts as a clamping vise, and the weight of the machine (about 175 lb.) eliminates vibration effects from the relatively light impact.

THE RELATION BETWEEN THE TENSION STATIC AND DYNAMIC TESTS[1]

By H. C. Mann[2]

Synopsis

Impact testing has been the object of intensive study both in this country and abroad for many years. From the earliest investigations of this subject, attempts have been made to correlate impact results with some physical characteristic of the static tension test but without definite results. In practically all these investigations, however, a wide variety of shapes and sizes of notched specimens was used, and since each gave a new series of impressions as to the interpretation of the results obtained, advancement to a thorough understanding of the true significance of the impact test was greatly retarded.

From the use of a tension form of specimen which could be tested under both static and dynamic conditions, data have been obtained which have led to the discovery of a correlation between these two types of tests. This correlation involves consideration of the fact that a material in its initial state possesses a certain amount of inherent internal potential energy which, during the process of deformation, is apparently converted to kinetic energy and manifested as increased elastic strength.

Data are presented regarding this internal energy factor which clearly indicate that the mechanism of the process of deformation and rupture is essentially the same under both static and dynamic conditions. It is also shown that since time is required for energy conversions to take place, the total energy value obtained must be dependent on force, time, and space, and therefore to obtain a complete knowledge of the dynamic properties of materials, the velocity of the force application must be considered.

In 1922 a most extensive and complete symposium on the impact testing of materials was presented before the Society[3] for the purpose of promoting a more definite and intensive study of the subject, to the end that the true significance and applicability of the impact test to problems of engineering might eventually be determined. This symposium, which may be taken to represent the present-day status of the impact test, clearly indicates the existing wide divergence of opinion as to its significance and interpretation, and quite definitely shows that results obtained are of value only to the extent that they may be used to compare certain characteristics of similar materials.

For the most part, the object of impact testing has been to obtain a certain result for one specific purpose which naturally led to the use of a wide variety of shapes and sizes of test specimens, each giving a new impression as to the interpretation of results. This chaotic condition has unquestionably been one of the greatest influences retarding advancement to a thorough understanding of the impact test.

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[1] Publication approved by the Chief of Ordnance, U. S. Army.

[2] Research Engineer, Watertown Arsenal, Watertown, Mass.

[3] Symposium on Impact Testing of Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 5 (1922).

Many attempts have been made to correlate results from the impact test with some physical property determined by the static tension test, as it is obvious that the discovery of such a relation would be of great practical value. While no definite correlation was obtained, these investigations brought out the following significant facts:

1. A material has the capacity of absorbing a limited amount of energy per unit of volume.

2. A material possesses a limited rate for maximum energy absorption.

The effect of volume on the total energy value obtained from the impact test is best illustrated by the tension-impact specimens shown in Fig. 1. These specimens were both 0.505 in. in diameter; one was 2.5 in. and the other 5.0 in. between shoulders. The volume participating in the absorption of energy in the larger one (represented by the length of uniform least diameter) was therefore twice that of the shorter. The 2.5-in. specimen absorbed approximately 1300 ft-lb. of energy and was ruptured. The 5.0-in. specimen absorbed the total energy capacity of the testing machine, 2170 ft-lb., and merely elongated but was not ruptured.

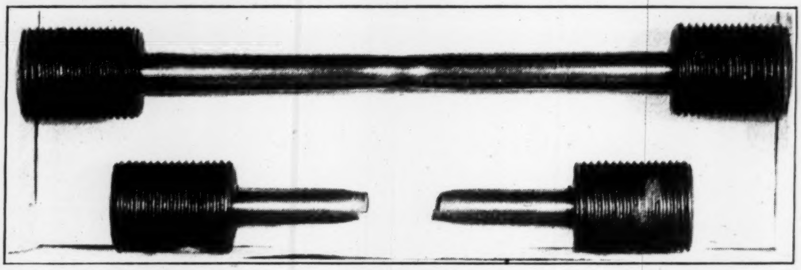

FIG. 1.—Tension-Impact Specimens Showing Effect of Volume.

This indicates in greatly magnified form that slight dimensional variations, particularly in the case of the notched type of specimen generally used, may result in considerable variation in the participating volume, and therefore in the total energy values obtained.

The effect of impact velocity on the resultant energy values will be considered in the latter part of this paper.

Considering the fact that from the impact test only a total energy value is obtained, it is evident that if a correlation is to be expected with the static test, a type of specimen must be used which can be tested under both conditions and yield the same essential data in each case. The type which lends itself most readily to these conditions is the tension form of specimen. This specimen can be made to such dimensions that the volume participating in the process of deformation can be easily calculated, and the elongation and reduction of area values readily compared under both conditions of test.

THE TENSION-IMPACT TEST

The tension form of impact test can be most readily carried out on the Charpy pendulum type of machine. In testing, one end of the specimen is screwed into the back edge of the pendulum, the other having been screwed into a cross-head or tup. When the pendulum reaches the lowest position of its swing, this cross-head strikes a pair of horns or projections attached to the base of the machine, stopping its forward movement and allowing the kinetic energy in the pendulum to elongate and rupture the specimen. The total energy value is calculated as the difference between the available kinetic energy of the swinging pendulum at the instant of impact, and the kinetic energy remaining after rupture. The particular

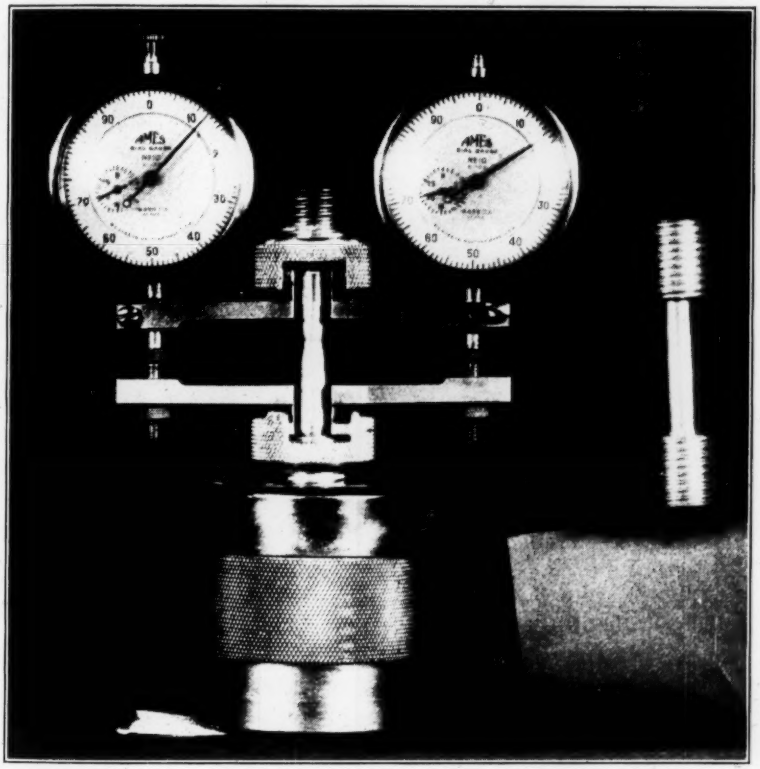

FIG. 2.—Extensometer and Specimen for Static Tension Tests.

Charpy machine used in this investigation has a capacity of 2170 ft-lb. which permits the use of the normal full-size tension specimens.

STATIC TENSION TEST

Since in the impact test only a total energy value is obtained, it is necessary to determine this same value from the static tension test. To do this it is customary to record each increment of load and corresponding elongation throughout the entire test, and from these data construct a curve the area under which can be evaluated in terms of energy.

In the usual tension form of specimen, the elongation produced by the applied load is measured over only a part of the total section involved and, therefore, the value determined from such a load-elongation curve will not represent the full amount expended in deforming and rupturing the specimen. From this it is evident that special consideration must be given to the development of both specimen and testing procedure, to insure that the energy value determined is fully representative of that actually expended.

The form of specimen and extensometer finally developed for this purpose are shown in Fig. 2. In this specimen the area of the gage section is one-third that of the end sections, which allows the specimen to be broken without stressing the end sections to beyond the elastic strength of the material. The elongation produced by the applied load up to the point of rupture is measured over the entire length of the reduced section by means of two dial gages of 0.0001 sensitivity attached as shown in Fig. 2, the readings being averaged to neutralize any possible effect of non-axial loading.

To obtain data from which the load-elongation curve can be constructed, the progress of deformation to rupture is carried out in a series of steps; the load is applied until the specimen has elongated a certain predetermined amount, at which point the loading is stopped until no further movement of elongation is noted. When this so-called balance is attained, the load and corresponding elongation values, as well as the change in diameter at the reduced section, are recorded. In this method of testing undesirable thermal effects produced during deformation are eliminated. A typical diagram plotted from data thus obtained is shown in Fig. 3. Since work is defined as force times distance, the integrated area under this curve, when evaluated as energy, represents the total amount imparted to the specimen, and as such may be referred to as external work.

When similar specimens are tested under both static and dynamic conditions, it has been found that the energy value obtained from the impact test is greater than the value of external energy as determined from the static test. Under both conditions, however, the specimen elongation and reduction of area values are similar, which would appear to indicate that the process of deformation to rupture was the same in each case. To determine the reason for this difference in energy values it is necessary to analyze the factors concerned.

It is a well-established fact that a specimen broken under impact is extremely hot at the instant of rupture, whereas when broken under static conditions very little temperature rise is noted, because of the much greater time allowed for its dissipation. To determine the amount of energy equivalent to the heat evolved during plastic deformation under both static and dynamic conditions, carefully calibrated fine iron and constantan wires were spot welded to diametrically opposite sides, at the middle of the reduced section, of the specimen and connected to a potentiometer. The temperature rise for each increment of deformation was determined from measurements of the changes in resistance produced.

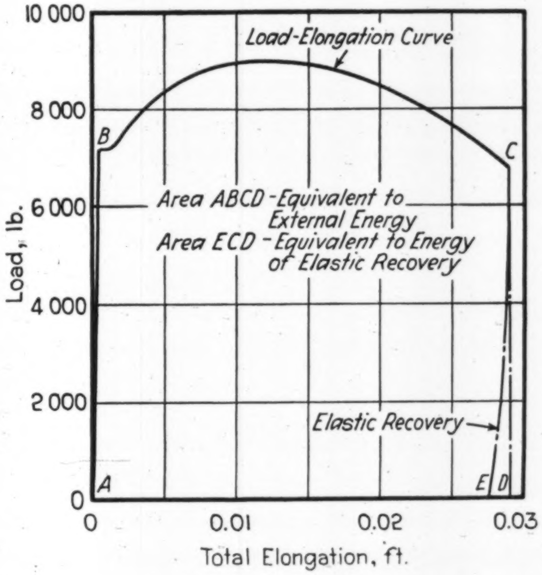

FIG. 3.—Load-Elongation Diagram.

In the static test the load was applied in steps as previously described, but in this case with sufficient rapidity that the potentiometer readings were obtained before the heat evolved had time to be dissipated. The total heat evolved during the entire test to rupture was obtained from the summation of that produced during each increment of deformation.

In the dynamic test the temperature rise was similarly obtained for various degrees of specimen deformation, as well as for complete rupture.

From the data it was found that the rise in temperature was inversely a linear function of the change in specimen area, and that the total heat evolved was approximately the same in both static and dynamic tests. Evaluated as energy, the total heat was found to represent about 90 per cent of the total value of external work, as determined from the load-elongation diagram of the static test. These results are similar to values obtained by Farren and Taylor,[1] who found that depending on the material, the evaluation of heat in plastic extension amounted to 86 to 95 per cent of the thermal equivalent of the work done.

In the process of deformation to rupture of a specimen, however, there are two additional effects which are also obviously concerned in the disposition of imparted external energy. These are, elastic recovery, and energy recovery manifested by the springing apart of both halves of the specimen, at the instant of rupture.

Considering a representative specimen used in this investigation, the value of external work was found to be 240 ft-lb. of which approximately 216 ft-lb. or 90 per cent was accounted for as heat. The energy equivalent of elastic recovery as determined from the area *ECD* in Fig. 3, was found to equal 2.4 ft-lb. or 1 per cent of the external energy value. The equivalent energy recovery value was determined from the amount of compression produced in a carefully calibrated copper cylinder 0.25 by 0.5 in. long placed in the testing machine in such a manner that it received the full force exerted by half the specimen when rupture occurred. From the amount of compression produced, the equivalent energy value for the whole specimen was calculated to be 22 ft-lb., or approximately 9 per cent of the total external work.

From this it may be concluded that in general terms the external energy is distributed as in the following equation:

$$\int \mathrm{d}Q = \int \mathrm{d}J + \int \mathrm{d}E + \int \mathrm{d}R$$

where $\mathrm{d}Q$ = external energy,
$\mathrm{d}J$ = thermal equivalent,
$\mathrm{d}E$ = elastic recovery equivalent, and
$\mathrm{d}R$ = energy recovery equivalent.

It has always been assumed that the total external energy as determined from the static test represented all of the work concerned in the rupturing of a specimen. There is, however, another form of energy conversion manifested during the process of deformation, which cannot be ascribed as a part of the external energy, and which has been entirely neglected in all previous investigations. This is the conversion of internal potential to kinetic energy, manifested as increased elastic strength.

A material in its initial state possesses a certain amount of inherent internal potential energy which, during the process of deformation, or cold working, is apparently converted to kinetic energy and manifested as increased elastic strength. This conversion of internal energy appears to take place only during plastic deformation, as it is in this period that the elastic strength of the material is increased. Since the increase in elastic strength is a direct function of the change in sectional area, the amount of the converted energy may be determined by obtaining from the change in sectional area at each increment up to the point of rupture the value of the load equivalent to the increase in elastic strength produced, and plotting these load values against corresponding values of total elongation (Fig. 4). The integrated area under this curve when evaluated in terms of energy is then equivalent to the total inherent internal potential energy of the specimen, and thus may be referred to as internal work.

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[1] W. S. Farren and G. I. Taylor, "The Heat Developed During Plastic Extension of Metals," *Proceedings*, Royal Soc. (London) A, Vol. 107, p. 423 (1925).

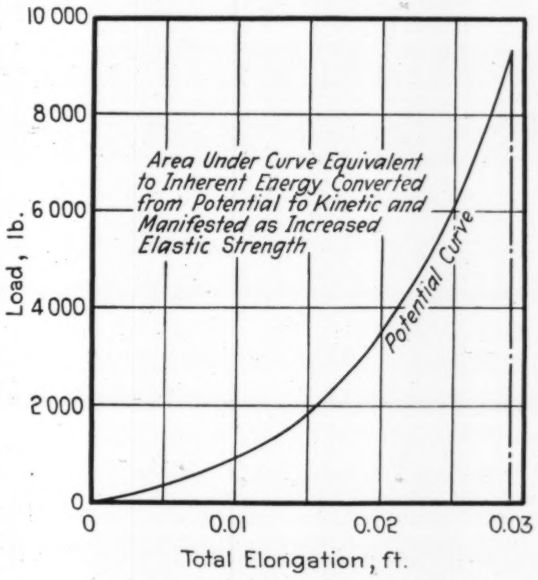

FIG. 4.—Potential Diagram.

The total work involved in the deformation and rupture of a test specimen must therefore include both external and internal energy values.

COMPARISON OF STATIC AND DYNAMIC VALUES

The results of a large number of tests under normal temperature conditions using similar specimens, of the form indicated, have shown that when the impact velocity is within a certain limiting rate, the energy value obtained from the impact test is equal to the sum of both the external and internal energy values as determined from the static test in the manner described. A typical example, and also a simple method for determining both these values from data obtained in the static test is shown in Fig. 5.

The so-called potential curve is plotted from data obtained by multiplying the load at each increment of deformation by the ratio of original to minimum reduced sectional area of the specimen at that point. The area between the potential and load-elongation curves is equivalent to the initial internal or potential energy of the specimen (the same as in Fig. 4). The area under the load elongation curve is equivalent to the imparted external energy. From this diagram the process of energy conversion can be readily visualized by noting that, as line AB moves to the right during the progress of deformation, it eventually assumes a position which may be designated as line GH. At this point the specimen possesses a less amount of potential energy than before, the difference having been converted to kinetic energy and manifested as increased elastic strength. As deformation progresses further the potential energy is gradually decreased by conversion, until the specimen, no longer possessing the ability to resist further deformation, is ruptured.

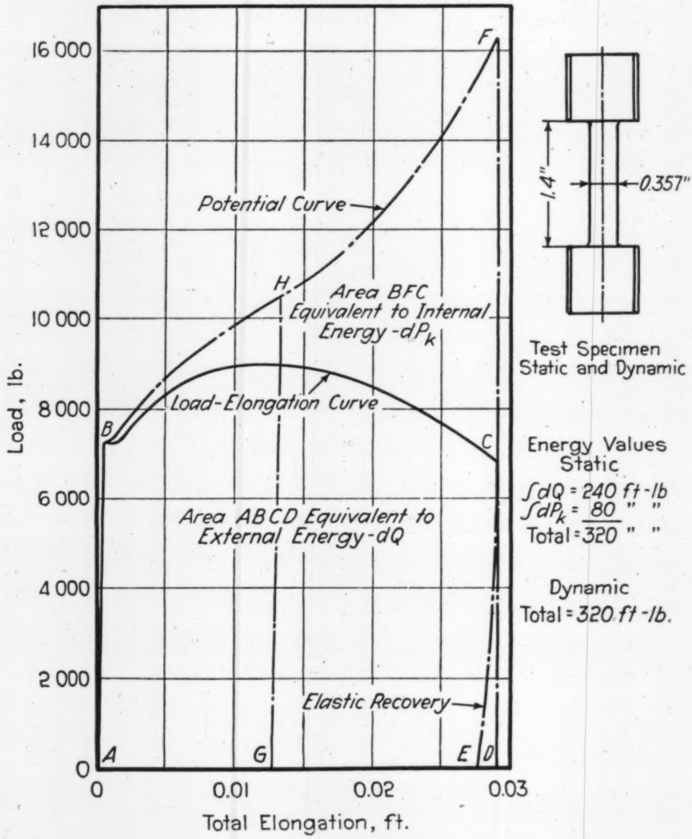

FIG. 5.—Total Energy Diagram.

Since test results have shown that the specimen elongation and reduction of area, thermal effects, and total energy values are practically the same under both static and dynamic conditions, it may be concluded that the process of deformation to rupture is also the same in each case, and therefore may be expressed as follows:

$$\int \mathrm{d}W = \int \mathrm{d}Q + \int \mathrm{d}P_k$$

where $\mathrm{d}W$ = total work,
$\mathrm{d}Q$ = external work, and
$\mathrm{d}P_k$ = internal work.

Typical values obtained from similar specimens of various compositions, tested at normal temperature under both static and dynamic conditions, are shown in the following table:

Gage Section of Specimen	Static Energy, ft-lb.			Total Dynamic Energy, ft-lb.
	External $\mathrm{d}Q$	Internal $\mathrm{d}P_k$	Total $\mathrm{d}W$	
0.357 in. diameter by 1.4 in.	260	105	365	362
	242	51	293	293
	207	50	257	257
	240	80	320	320
	304	151	455	454
	262	92	354	354
0.505 in. diameter by 2.5 in.	734	260	994	994
	625	188	813	814
	708	240	948	946
	694	253	947	947
	750	256	1006	1003
	983	260	1243	1250

EFFECT OF VELOCITY

The effect of velocity on the energy value obtained from the impact test has received comparatively little attention in the past. While it has been definitely shown that certain materials are sensitive to velocity changes,[1] progress in this direction has been limited because of the fact that, without the value representing the maximum energy conversion property of the material at zero velocity, obtainable only from the static test as previously described, any possible effect of velocity on the impact value could not be determined.

With most materials it has been found that when the impact tests are conducted at the normal velocities of the usual commercial types of machines, from about 11 to 17 ft. per sec., the energy values obtained are practically always equal to those determined from the static test in the manner previously described. When the impact velocity is increased, however, the impact values, and also the specimen elongation and reduction of area values, are found to be considerably reduced. Typical examples of the difference in values obtained from the static or zero velocity test, and the impact test at 28.5 ft. per sec., are as follows:

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[1] R. G. Batson and J. H. Hyde, "Testing of Materials of Construction," Vol. 1, p. 310, E. P. Dutton and Co., New York City (1922).

	Total Energy, ft-lb. (d*W*)	Elongation, per cent	Reduction of Area, per cent
Static	373	24.2	66.0
Dynamic	301	21.0	62.6
Static	356	23.6	64.7
Dynamic	278	20.0	60.2
Static	365	23.6	63.4
Dynamic	308	20.0	60.6
Static	1225	80.0	83.0
Dynamic	664	58.0	69.0

To determine the trend of impact values, a few steels which exhibited this velocity effect, and one which showed no change, were tested at velocities ranging from zero, of the static test, to 28.5 ft. per sec., the change of velocity being obtained by varying the height of fall of the pendulum of the Charpy impact machine. The results obtained from these tests are shown in Fig. 6.

These curves clearly indicate that the complete dynamic properties of materials cannot be definitely determined from the usual type of impact test carried out at only one single velocity.

Discussion

It has been shown in the data presented that when similar specimens of the same material are tested at normal temperature, under both static and dynamic conditions, the total energy values obtained from each are the same, provided the velocity of impact is within a certain limiting amount, and that when this velocity is exceeded the impact values become considerably reduced. A possible explanation of this phenomena is obtained from a further consideration of the mechanism of the static test.

In the static test, the force as it is applied to produce each increment of deformation causes a change of state in the material which, during the process converts a certain amount of the inherent potential to kinetic energy, resulting in increased elastic strength which is just sufficient to resist or balance the applied force at that point. As the applied force is increased, this change of state and energy conversion continues until eventually a point is reached where all of the inherent potential energy has been converted, and therefore, since no further resistance can be offered, rupture takes place.

During the process of each increment of deformation, there is always a certain time lag between the force application and the resultant increase in resistance produced. Thus, when the applied force is continuously increased, the full resistance or balance is never completely attained and, therefore, deformation proceeds until rupture occurs. If at any increment further deformation is to be prevented, the applied force must be reduced to the point that it will just balance the resistance offered.

In the impact test the total energy value obtained is customarily referred to as the energy absorbed by the specimen in undergoing deformation to rupture. From the viewpoint of the correlation between static and dynamic tests, this conception is somewhat misleading in that it gives no indication of the fact that, since the mechanism of the process of deformation has been concluded as the same in both types of tests, the total impact value must include both the work of deformation and inherent energy conversions. Considered in this manner, the total energy value obtained from the impact test assumes a far greater significance.

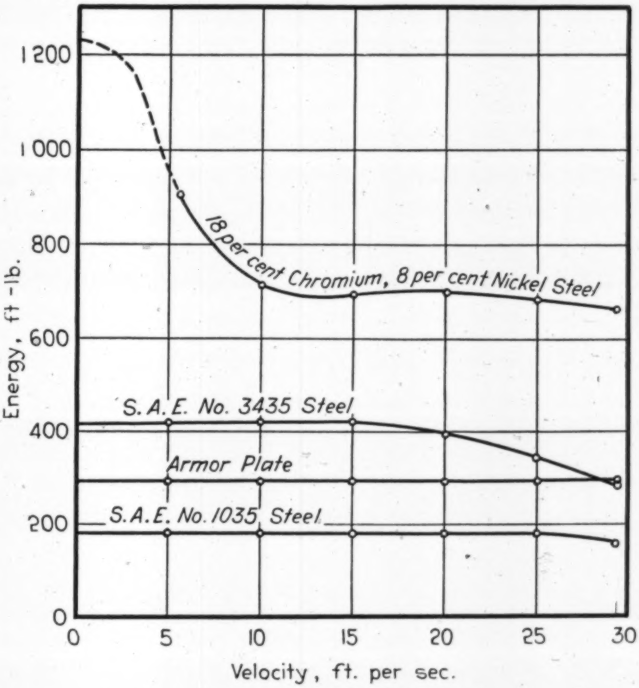

FIG. 6.—Showing Effect of Velocity.

These considerations imply that the work involved in producing deformation must be independent of the magnitude of the applied force, and that a definite period of time, dependent on the material and condition, is required for the internal changes to take place during deformation.

From these assumptions it is evident that when similar specimens from the same material are tested under both static and dynamic conditions, the total energy values obtained in each case should be the same, provided that the impact velocity is within the limiting rate for maximum deformation and energy conversions to take place. As the impact velocity is increased, however, the impact value as well as the elongation and reduction of area values should be correspondingly decreased. That these statements are correct is amply substantiated from the results of a large number of tests.

In the determination of a correlation between the static and dynamic tests, it has been deemed essential to consider only these factors: temperature, increased elastic strength, elastic recovery, and energy recovery, which could be definitely evaluated and shown to be concerned with the final results.

While it is recognized that there are undoubtedly other forms of energy conversion and distribution taking place during the process of deformation, they apparently cannot be actually measured, and since no material is perfectly isotropic or homogeneous, these changes must be not only of a variable nature, but also of such minor significance that it appears entirely reasonable to consider them as merely effects produced by the major factors referred to.

Although the data presented were obtained from what may be considered as the long-notch form of tension specimen (Fig. 5), this same correlation between static and dynamic tests has been found to apply in the case of similar specimens having notch lengths as short as 0.03 in. From a series of experiments now being conducted, it has been tentatively determined that as the notch length of the test specimen is shortened, the following general effects are produced:

1. The specimen becomes much more sensitive to the influence of velocity.

2. The total energy value decreases in a linear relation to the volume and reaches a minimum when the notch length is equal to approximately one-third the diameter of the reduced section.

It has been found that to obtain a correlation between the static and dynamic tests, it is essential that the form of the test specimen be such that the full section participating in the deformation and conversion of energy can be readily measured. This necessitates square or parallel shouldered notches as indicated, since if the shoulders are tapered or otherwise formed, deformations occur in these sections which will not be included in the determination of the total energy value of the static test. Under such conditions, the static obtained will always be less than the dynamic value, thus leading to possible erroneous conclusions regarding a correlation between the two types of tests.

CONCLUSIONS

1. The mechanism of the process of deformation and rupture is essentially the same in both static and dynamic tests.

2. The work involved in producing deformation depends on force, time, and space, and therefore must be independent of the magnitude of the applied force.

3. The rate of deformation for maximum energy conversion is dependent on the composition and condition of the material.

4. The dynamic properties of a material are dependent on three major factors; volume (as determined from the length of uniform least diameter or section), velocity of the applied force, and material condition (also influenced by temperature).

5. The present available types of impact machines are too limited with respect to velocity for determining the complete dynamic properties of materials.

While the data presented concerns only the tension form of test specimen, it appears entirely reasonable to assume that the theory is equally applicable to other types as well. In the case of the transverse notched type of specimen, the determination of inherent energy conversions are obviously a practicable impossibility, and while it might appear that this particular form of specimen reveals to a better advantage certain undesirable material characteristics, this is apparently due to the fact that with the form of notch used greater sensitivity to velocity conditions is obtained. However, this condition should be equally indicated in the tension form of specimen when tested at the higher velocities.

To obtain a complete knowledge of the dynamic properties of materials, impact tests should be conducted through a range of velocities of a magnitude sufficient to definitely reveal material embrittlement. The combination of such data with that of the static test, should make possible definite predictions as to the subsequent behavior of materials under known service conditions.

DISCUSSION

Mr. P. G. McVetty[1] (*presented in written form*).—A study of this paper shows that much of the confusion which results from attempts to interpret and correlate impact tests may be due to the very complicated stress distribution in the usual notched transverse specimen. The close agreement between static and dynamic energy values seems to indicate that the tension impact test may now be interpreted in a logical manner.

I was particularly interested in the velocity effect. It is becoming more apparent that speed of testing has a far greater bearing on many of our tests than has been realized. It has not been customary to specify a definite speed for impact tests and this work shows clearly that it would be very desirable to make further investigation of the effect of this variable.

The author has mentioned the effect of temperature on impact values and it occurs to me that a study of this kind at various temperatures would yield some valuable data. It is hoped that he may continue to make further investigations of this type and that others will give more thought to the interpretation and correlation of test data.

Mr. M. O. Withey.[2]—In this paper the author makes some statements about the total energy of the specimen and indicates that it is proportional to the volume of the test piece between the shoulders. In our work at the University of Wisconsin, we have, for a number of years, made impact tension tests but we have always evaluated the results in terms of unit volume. I believe that it would be preferable in reporting data of this type, to report it in inch pounds per cubic inch, basing the volume of the material on the length of the test specimen between shoulders. By adopting this procedure small differences in the length or diameter of the specimen would not affect the results and if different investigators did not use exactly the same specimens, the results would be approximately commensurable. If, on the other hand, total energy is reported, it becomes necessary to reduce this information in terms of unit volume.

Mr. H. F. Moore.[3]—These results, of very great interest, have been applied so far to ferrous metals, and our general experience with some of the non-ferrous metals is that the laws of strengthening by cold working do not seem always to be quite the same, and I hope that Mr. Mann or others will carry on this work with the non-ferrous metals.

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[2] Professor of Mechanics, University of Wisconsin, Madison, Wis.

[3] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

MR. W. W. WERRING[1] (*by letter*).—The agreement shown between the energy values for the dynamic test and those of the static test when calculated by the method described is certainly startling. However, the explanation offered in terms of an inherent potential and kinetic energy of the specimen itself leaves something to be desired.

The basis of this explanation is the concept of an "internal potential energy of the material" which is converted into kinetic energy. In the first place, it is difficult to associate the internal changes of the specimen with the terms potential and kinetic energy as commonly used. Secondly, if we accept these explanations of internal energy conversions at face value some explanation is required to justify the arithmetical addition of the "converted energy" to the externally applied energy in the static test. It does not appear sufficient merely to say that the total energy must equal the sum of internal and external energy and thereupon add numerically. Unless we justify the choice of algebraic sign of the various energy quantities we may be doing something akin to adding "action" and "reaction" to determine a total force. In fact, the addition of the specimen's energy to that externally applied by the machine implies that the specimen by its internal energy aided in its own breaking.

When we consider the case of the dynamic test, the explanation also seems inadequate. In effect, it is merely stated that with the pendulum impact machine the indicated energy is the total of both internal and external energies. That energy applied externally to the specimen by a pendulum machine is less exclusively external than that applied by the slower moving tension testing machine appears to require further explanation.

I am anxious that this discussion of Mr. Mann's suggested explanation of the relationships he has observed should not detract from the importance of the discovery of the relationships themselves. I hope Mr. Mann will publish additional detailed data in order thoroughly to establish the foundation of facts as a basis for further study. I am unable to advance a better explanation than the author's, but it might be well to observe that the curve which he shows as the "potential energy curve" is the well-known "real stress" curve computed to load values. In this connection, it would be interesting to know whether materials in which the real and engineering stress curves are closely alike show the same energy values in the static and dynamic test. Such materials, of course, are brittle materials which fail without reduction of area.

MR. H. C. MANN[2] (*author's closure by letter*).—In view of the fact that the theories advanced in this paper present a somewhat radical departure from established ideas regarding the subject of impact testing, the interest evidenced is most gratifying.

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[2] Research Engineer, Watertown Arsenal, Watertown, Mass.

It seems to me that much of the confusion now existing in the interpretation of the impact test is the result of attempting to apply elastic stress theories to conditions involving plastic deformation, rather than to any very complicated stress condition as suggested by Mr. McVetty. The change from one condition of elasticity to another by means of plastic deformation, appears to be accomplished only through some form of internal energy exchange which must be considered entirely separate from any elastic stress condition. Tests which we have carried out at velocities up to 200 ft. per sec. have shown that the mechanism of the process of deformation to rupture, as well as the actual stress conditions of the material during this process, are the same under both static and dynamic conditions, which further emphasizes the fact that consideration of energy rather than stress is the important factor in interpreting the impact test.

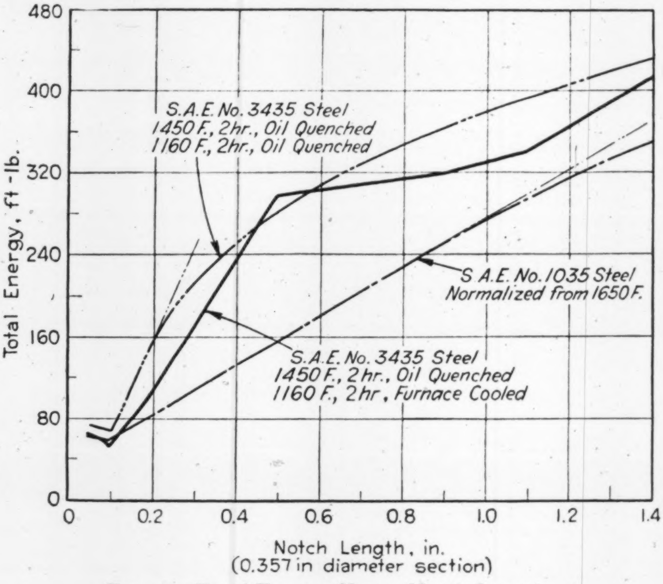

FIG. 1.—Total Energy *Versus* Notch Length.

As to the velocity effect, I thoroughly agree with Mr. McVetty that the speed of testing is a most important factor which has been heretofore almost entirely neglected. To obtain further information regarding speed of testing we have designed and built a variable speed tension impact machine having an ultimate capacity of 1000 ft. per sec. velocity. Tests to date at velocities up to 200 ft. per sec. have shown extremely interesting and rather startling results. The data however are insufficient to report at this time.

Referring to Mr. Withey's remarks, the reason for using total rather than unit energy values was to bring out more clearly the correlation between static and dynamic results. The practice of evaluating energy in terms of unit volume would be perfectly satisfactory provided all specimens tested were of the same dimensions. We have found that when the dimensions and total volume are changed, the unit values are no longer constant. This is quite clearly shown in the accompanying Figs. 1 and 2, which indicate the total and unit energy values obtained from tension impact specimens of various volumes. While only three curves are shown, we have found this condition to apply in all materials similarly tested.

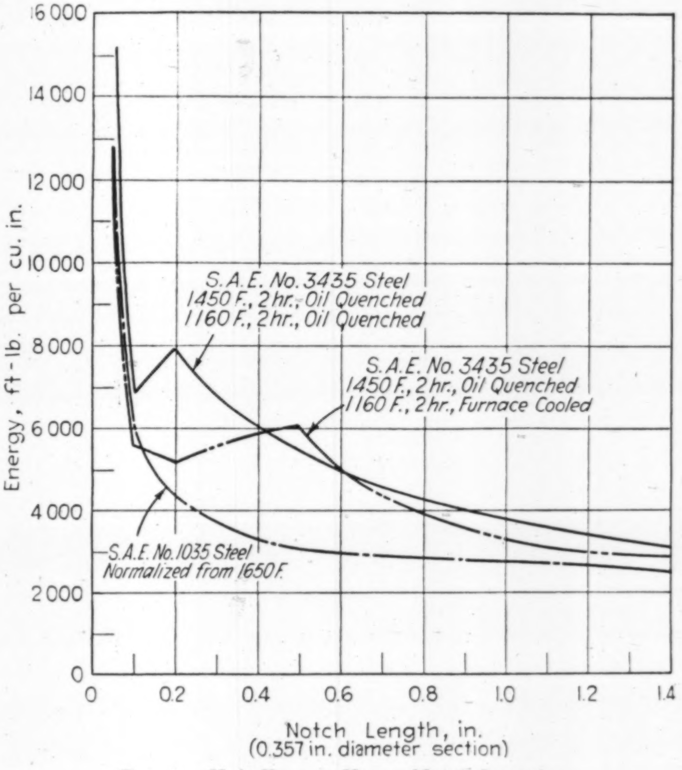

FIG. 2.—Unit Energy *Versus* Notch Length.

In reply to Mr. Moore, we have carried out similar tests using specimens of the various classes of bronze, brass, monel metal, aluminum, etc., and in every case have found the same correlation to hold. Although not so thorough a study has been made of the non-ferrous materials, we have yet to find evidence that the theories advanced are not as equally applicable as they are to the ferrous metals.

The comments of Mr. Werring are most difficult of clarification. While it is quite evident that no one really knows just what occurs within a material when plastically deformed, it seems entirely reasonable to assume that from the fact that a material offers resistance to plastic deformation, there must be present some form of inherent internal energy which affords this resistance. Also, when plastic deformation occurs, there is a change in shape as well as in physical properties, and furthermore, less work is required for rupture after such deformation than before, all of which seems to indicate that deformation produces a change in internal energy conditions. In the static test it is customary to measure the increments of force-distance movements and evaluate them in terms of total energy. This would be a perfectly correct procedure if the specimen did not plastically deform, but the fact that deformation takes place infers directional movement in other than the line of force. This movement, which may be considered as radial since deformation occurs in that direction, does not enter into the normal energy calculations. It therefore appears that in considering this radial movement, which is done when the applied force is increased in the ratio of original to reduced area, the relative values of the internal energy changes are obtained.

In the dynamic test the value customarily obtained is energy, and not force or stress. Tests which we have carried out on carefully prepared specimens broken at velocities up to 200 ft. per sec. have shown that the maximum stress sustained by the specimen during rupture is the same as in the static test. It therefore seems clearly evident that the commonly applied force - total elongation method of evaluating energy in the case of the static test where radial deformation takes place is fundamentally incorrect.

While the terms "internal potential" and "internal kinetic energy" may not be the most applicable for the purpose, they seemed to be the best suited to indicate the energy conditions which it appeared reasonable to assume exist.

As to the "potential energy curve" being the same as the "true-stress curve," this is only natural since they both represent actual elastic strength conditions existing in the material during the progress of deformation to rupture.

In answer to the question regarding static and dynamic values of so-called brittle materials, we have tested 0.95 per cent carbon steel which had an elongation of only one or two per cent as well as other materials of similar characteristics, and have found the same correlation as with the more ductile materials.

A SURVEY OF TESTING IN THE PRECIOUS ALLOY FIELD, WITH SOME NOTES ON THE TESTING REQUIREMENTS FOR DUPLEX ALLOYS

By Thomas A. Wright[1]

The eight metals termed "precious" are silver, gold, platinum, iridium, rhodium, palladium, osmium and ruthenium. They are employed chiefly in the wrought condition and in alloy form. Articles in the form of castings are rare. One of the most common uses is as an electroplate, finish or as it is at times termed, "coloring."

While appearance and cost are major factors in the choice of any one of these metals, this paper approaches the subject primarily from the viewpoint of utility. That is, an attempt is made to survey the field of application only to the extent of emphasizing those classes for which specifications have been, or are likely to be, written. The engineering features of these specifications on the whole have until recently been neglected, and legal, commercial, and chemical aspects have been foremost.

Even silver, the least expensive of the group, is sold on the basis of ounces, rather than tons or pounds, while gold and platinum are often sold by the pennyweight. Because of the high monetary value, older specifications, such as National and State stamping acts and laws(**3-5**),[2] have been written around quality and marking provisions. More recently trade practice provisions written into certain codes of fair competition have accepted the principle of a minimum quality of precious metal content as well as requiring fairly definite statements as to the amount present.

Gold is in common use today even in highly utilitarian articles. Such articles, contrary to former practice, are not always made by craftsmen, artisans or artists but by mass-production methods which have called first for the services of the mechanical engineer, and more recently, of the metallurgical engineer or chemist. Volume production and constant use have given rise to problems of quality, strength, ductility and corrosion. Into all of these, testing enters.

The metals are employed in many forms: as pure or commercially refined metal, as alloyed with one or more other precious metals, as alloyed with one or more base metals either by melting or electro-deposition, or as duplex metal. Compressed powdered precious metal articles are in the offing.

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[1] Technical Director, Lucius Pitkin, Inc., New York City.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 362.

USES AND APPLICATIONS

The applications of precious metals may be grouped according to purpose, as, for example, investment, decoration or utility. For the first of these, testing would be primarily for the purpose of ascertaining the basic monetary value and quality, with special reference to coinage acts and currency regulations. For the second, testing would be for purposes of inventory, production control and conformity to provisions of the National Stamping Act, etc. For the third it would be for conformity to the value and formula provisions of the specific purchasing specifications, resistance to corrosion, and compliance with the necessary physical requirements. Or again, they may perhaps be better divided into groupings more indicative of the character of the article; thus economics, industrial, household, personal adornment and use. This grouping appears to afford a better survey, the object of which is to indicate the fields in which A.S.T.M. functioning would be of the most economic value.

Economics:

As bullion or in fabricated forms, silver and gold in particular, and to a lesser extent platinum, have been employed for centuries by most governments as convenient means of investments or for purposes of monetary reserves. Such use by individuals has been confined chiefly to those of oriental or Levantine origin and to a somewhat less extent to some of the Latin races. As bullion, the metal may be largely in commercially refined or elemental form; as currency, gold and silver are invariably in alloy form, the non-precious metal added being copper. As a rule, currency is tested for fineness (parts per thousand) only—except for numismatic purposes—and the testing is invariably carried out by a government mint or assay office for government control or purposes of exchange. More rarely, sales may be made of coins to a smelter or refinery, as when a currency is called in due to a change of policy or absorption of one nation by another. Or again, as recently, when a change in a governmental policy or relationship permits of a refining and resale profit on either bullion or coinage.

The methods of assaying are substantially standardized. They, like the provisions as to gold or silver fineness of coins, are not within the province of the A.S.T.M.

Industrial:

The chief industrial uses are in the electrical, chemical, automotive and photographic fields.

Contact points and thermocouples comprise the major forms in the electrical field. Contacts may be made of platinum or platinum-iridium, copper-palladium, silver-palladium, silver, silver-copper, or even at times, white gold. A platinum type is used in high-tension sparking and a silver alloy for less drastic requirements or in temporary or less costly equipment as in signaling, detection or automotive work. Chemical or spectrographic analyses are made for percentage of major elements, alloying constituents and impurities that might result in pitting or in an oxide film that would reduce conductance. Physical tests cover resistance to wear-corrosion, electrical resistance or conductivity. The latter tests may depend upon the character of the thermostat or spring-arm base alloys to which they may be riveted, welded or soldered. Thermocouples of the precious metal variety are generally made of platinum alloyed with rhodium although at one time platinum-iridium was used. Working specifications and tests for such articles fall within the province of the Society.

For chemical engineering uses, platinum and silver, the two of importance, are employed as metals, the former in decreasing and the latter in increasing amount and proportion. Platinum of commercial purity finds its chief application as a catalyst and as such may be eliminated from further discussion here. Silver, because of exceptional corrosion resistance to fruit acids and alkalies, is finding steadily increased use as fabricated vessels and piping, particularly in the beverage and food industries. Its peculiar bactericidal properties are just beginning to be applied, for example, in the sterilization of water. The chief need for specifications and testing lies, however, in the field of chemical apparatus where resistance to corrosion, temperature, and pressure requirements vary. Problems of forming and fabrication also involve chemical and physical tests.

In the automotive field, magnetos being *passé*, only silver is of interest. Its use in the electrical system has been mentioned. The only other important one is that of a "finish" on the hardware, where it is employed for decorative or protective purposes in the form of an electrodeposit (sometimes lacquered). The chief tests would be for distribution, uniformity, thickness and weight of coating. Such tests are certainly within the province of the Society. Silver in bearing metals is just coming into use.

In the photographic field, only silver is of any importance. Gold and platinum find a limited use in prints. The silver used is in the form of the refined metal, the tendency being strongly to as highly purified metal as may be obtained. Due to the small number of companies involved, there is no demand for general specifications. The indications are, however, that the normal methods of testing refined silver may not be sufficiently precise. In other words, refined silver is normally tested for silver content expressed as fineness. It may be necessary to approach the question of composition from the other direction—that is, determine the nature and amount of the impurities. This is still another example of the necessity for defining, with increasing care, the use of terms such as "refined," "chemically pure," "spectroscopically pure," etc.

Solders, of course, are used in many fields but from the Society's point of view utility is of more importance than art. Solder as an industrial item is of major importance so far as the silver type is concerned. Silver solders, however, have been ably discussed by Leach(1) and specifications set up(2). Gold solders are used chiefly in the jewelry and dental fields. The dental type in particular may contain platinum or palladium or both and has been the subject of some research due to the rigid corrosion-resistance, fatigue or strength requirements when in use. In this field there are no standardized formulae or methods of test, except those developed at the National Bureau of Standards for the American Dental Association.

Household:

The form of articles for household or commissary use is usually one of utility and convenience whatever the metal or decoration employed. Cost being a major consideration, articles of gold (and platinum) being luxuries, may be omitted here. Attention is directed rather to the only real metal of importance, silver, which is employed either as solid or plate. In the United States, solid silver, or sterling silver, as prescribed by the National Stamping Act(3) consists of an alloy of silver and copper in the proportion of 925 parts silver to 75 parts copper. For all practical purposes all other ratios may be disregarded. Other alloys of lower silver content have been developed and by suitable propaganda may find a more or less wide use but it will be because of superior tarnish-resistant qualities or some similar reason, and such other alloys cannot be termed "sterling." Susceptibility of sterling silver to tarnish when exposed to hydrogen sulfide has encouraged the use of chromium plating on articles for personal use. Latterly, this has been superseded by rhodium plating. Neither thickness nor measurement thereof has been subject to specifications. The percentage (fineness) of silver in sterling is checked as a rule by established fire-assay methods, at times a correction (usually of the order of 1 per cent) being made for volatilization and slag losses by running a proof.

The greater proportion of flatware and tableware is electroplated. Silver-plated articles consist almost wholly of an 18-per-cent nickel-brass or similar alloy base electroplated with commercially pure, or as it is known, "fine" silver. Certain designated marks may at times denote the quality in terms of pennyweights per piece or per dozen or, as is now specified for flatware in the code(4) for the silverware industry, troy ounces per gross. Provisions are also made for the marking of spoons, for example, where extra silver is deposited on the parts subject to most wear. No method has been established for ascertaining the weight of the deposit or the thickness and character of the reinforcing or inlay. Neither is any provision set up as to the method of selecting the samples to be tested or the number. In view of the large purchases made by hotels, railroads, steamship lines, etc., a standardized testing and stripping procedure would appear to be in order for tableware at least, whether or not such code provisions are retained in some other form.

Ornamental pieces may be either solid or plated and as a rule are coated with a clear lacquer. On plated articles, the base, if domestic, will usually be a nickel-brass, but modern items, such as book-ends and candlesticks, if of oriental make, may be of low-grade brittania or even of hard lead. The silver is usually deposited on the latter on an intermediate plate of nickel or even nickel on copper. Due to the operation of present Treasury Department regulations under the present Silver Act certain tests essentially of a semi-quantitative nature are necessary for customs purposes. Here the spectrograph, microscope or assaying all come into use.

Personal Uses:

Silver at the moment is in vogue for sterling silver jewelry with a rhodium finish. Comparatively little mechanical or electro-plate is used. Some sterling silver plated with a gold alloy is being produced.

Gold finds many applications of a utilitarian as well as personal nature. The uses as jewelry in the solid form, while governed by stamping acts and laws, are not usually of a character to require other specifications. Determination of the gold, etc., is by assay. The other chief industries employing this metal in some form are optical, watchcase, and pencil and pen. In these the great bulk of production is in the form of duplex alloys, known generally as "plate," in which gold is kept within certain designated ratios. Gold is alloyed with other metals either by melting or by electro-deposition. It is rarely used in pure or unalloyed form even in jewelry. Fine gold or 24 K is employed for gold leaf, but even here 20 K and 22 K alloys are common, especially for decorative purposes where large areas are to be covered.

A minor use, again in alloy form or as an electro-plate of pure gold on silver or a base metal, is that of lace, braid and buttons. Here testing for quality or gold content and also as to method of manufacture is practiced although no testing specifications of an official character are known to the author. Testing, however, is not uncommon and is increasing and the U. S. army and navy buy on specification.

Karat gold alloys (miscalled "solid gold") are usually made in four colors. Regular gold, shading from red to yellow is an alloy with copper and silver to which zinc and more rarely nickel are at times added. Green gold is a somewhat similar alloy, while white gold—now lessening in favor—has a base of copper, nickel and zinc and more rarely may carry some palladium. Such colors are common in the jewelry, watchcase, and pencil fields. On the other hand, the optical industry in addition to white prefers a color which is relatively inconspicuous and is variously known as pink, coral or tan. All of these alloys may at times be analyzed. This is especially true of dental alloys used only as "solid" and often containing platinum and palladium.

The bulk of the "karat gold" alloys has been of the 10, 12 and 14 K grades with some 18 K. An unwritten law has restricted production in lower karats but the recent rise in the price of gold has tended to encourage production of 8 and 9 K goods.

Plated gold is made by several processes. The bulk of what is known as "low-grade" and "medium-grade" jewelry and allied products such as cigarette cases, compacts, etc., is of this type. Most of the past production, except in the cheapest grades, has been made by what is known as "the rolled-gold plate process." The use of that term and of "gold-filled" is today restricted in addition by a Department of Commerce(6) trade-practice standard and by certain codes. Such plate is made by soldering or welding a strip of gold alloy to a thicker stock of base metal or alloy. For watchcase purposes the ratio of the former to the latter is by thickness but in the jewelry, optical and pencil fields by weight.

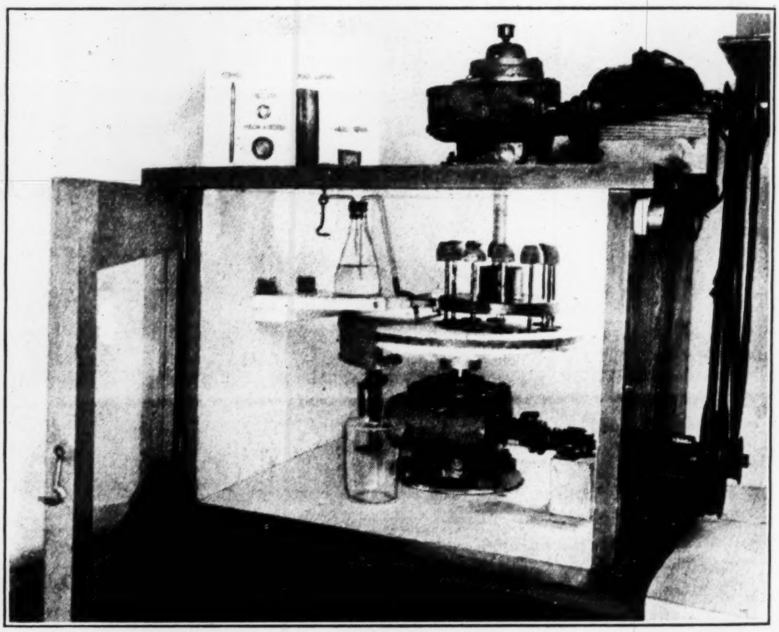

FIG. 1.—Front View of Pitkin Wear-Corrosion Testing Apparatus.

A more recent innovation is the electro-deposition of the gold alloy followed by a suitable heat treatment. This in turn has encouraged new life, fortunately accompanied by considerable improvement in quality, for the old, simple electroplate. The latter is still used to give a "finish" or "color," sometimes for artistic purposes, sometimes for other purposes.

Testing, in order to differentiate the several types and also to determine karat, thickness, ratio and corrosion resistance, is fortunately becoming somewhat more common. Certain phases of testing in this very important field are discussed in a later portion of this paper.

Tarnish and corrosion testing have been applied, usually using sulfur compounds or fruit juices. More recently wear-corrosion tests employing synthetic perspiration have been carried out and an apparatus therefor developed in our laboratories. The Pitkin Wear-Corrosion testing apparatus (Fig. 1) consists in essence of an upper and lower round plate revolving on the planetary principle. The upper or smaller plate is fitted with 12 holes through which pass as many spindles rounded at the lower end to engage a socket in the upper face of the small 2-in. disks to be described later. The upper end of the spindle may carry loads of 2, 4 or 8 oz. as desired. The back of the sample, which may be, for example, in the form of an electroplated half-round ring, is cemented to the lower face of the brass or other metal disk. The lower or front side of the sample then rests face downward on a suitable polishing cloth clamped by means of a ring to the lower revolving and larger plate.

The motive mechanism having been started, the upper plate moves clockwise and the lower anti-clockwise. The axis of the upper plate being located on a somewhat higher plane at a point midway between the periphery and axis of the lower plate, the movement is a planetary one. Pitting is thus minimized. As the upper plate revolves it is arranged to automatically operate a trip mechanism, thus delivering measured quantities of the corroding medium which may be synthetic perspiration, a fruit juice or what-not. The report of examination may be based on time or number of revolutions required to cause failure.

In some cases carefully controlled abrasive material has been employed in order to adapt the tests to the harder electroplates, such as chromium and rhodium, and also to accelerate the test. Specimens are best tested in multiple and several comparison sets may be run simultaneously. This type of test, coupled with microscopic studies for distribution and thickness, is particularly suitable for any article worn or used in contact with the skin.

Platinum alloyed and hardened with 5 to 10 per cent of iridium is employed in the solid form. The use of platinum "top" or plate is nearly obsolete because of the difficulty of properly controlling the marking and stamping of such jewelry.

Articles for personal use may be classed either as those for adornment or for utility. Platinum pieces are confined to the first group. Assaying of finished or even semi-fabricated stock for consumer purposes is not very common. There are no standard specifications for platinum although certain grades are accepted. In the main, the chief reliance of the manufacturer is on the reputation of the supplier of raw material. Most articles are bought on faith. Some have been sold on hope. Charity may say no more.

Palladium, as such, is not used for fabricated articles but as stated previously is an important constituent of some dental alloys and offers certain possibilities as metal or a silver alloy when made up in the form of plate. Methods of analyses of high precision as published by the National Bureau of Standards for the whole platinum group are nevertheless time consuming and costly. Short methods are desirable but are hardly of sufficient importance to warrant extensive governmental expenditures, when compared with other public needs.

Osmium and ruthenium may be disregarded because of limited production and use. Rhodium as an electroplated finish has been considered. It remains only to mention that in assaying it will, to a varying extent, remain and be weighed as gold. This at times necessitates a correction therefor.

SUMMARY

Only the more important precious metal forms have been discussed. An attempt has been made to outline the types of tests required. Judging from the author's experience and that of his organization in this special field, the order of importance from the point of testing would be gold, silver, platinum and rhodium. The tests to be applied include—again in order of present trade practice—assaying; analysis for formula; studies involving identity, character, thickness and distribution of plate and electroplate; tarnish, corrosion and wear-corrosion; hardness; and ductility, springiness, etc.

It is hoped that the foregoing survey may serve as a background by which the question of specifications and testing of precious metals may be approached.

One such problem will now be discussed as it is one of real importance.

CERTAIN TESTING REQUIREMENTS FOR DUPLEX OR MULTI-METAL PRODUCTS

The title of this portion of the paper was difficult to choose for the materials dealt with include not only rolled-gold plate, gold filled, heat-treated electroplate (a development of rather recent origin referred to commonly as Bekplate) but jewelry, optical, watchcase and platers scrap.

Gold-filled and rolled-gold plate were defined in 1933 in a trade practice or Department of Commerce standard(6). By this agreement the use of the two terms is restricted, as follows:

> "The quality mark 'Gold Filled' and/or 'Rolled-Gold Plate' shall refer to articles made of base metal, upon one or more sides or surfaces of which base metal there is affixed by soldering, brazing, welding, or other mechanical means, a sheet or sheets or shell of karat gold, produced by alloying fine gold with other metals, said sheet or

sheets or shell of gold being rolled, drawn, or pressed to the marked weight ratio before uniting with the base metal."

On the other hand the code for the watchcase industry(7) uses a somewhat different wording. There are no specific standards for electroplate whether heat-treated or not.

Five main factors have served to bring testing to the fore:

1. The provision of the new Department of Commerce standard(6) which permits of a tolerance of 10 per cent from the marked quality of articles (watchcases do not come under this standard) is interpreted in some quarters to apply only to the manufacturer who fabricates, solders, assembles and polishes the finished article, in order to cover such operations. The point is made that the manufacturer of rolled-gold plate must supply "plump" or full quality stock to the jewelry manufacturer. Quite obviously the rolled gold plater is entitled to credit in the "quality" assay for every last grain of gold.

2. The introduction of quality standards in trade-practice clauses of codes(10) and increased interest in trade and consumer circles.

3. The introduction of heat-treated electroplated gold alloys on a base-metal foundation.

4. The increased use of nickel and of nickel-silver of higher nickel content. Increased nickel content increases the viscosity of the slag and raises the melting point of both metal and slag. This has resulted in assay figures lower than are often realized, unless the assayer has taken the necessary precautions.

5. The recent increase in the price of gold to some 70 per cent over the old statutory price of $20.67.

Metallographic:

There is accordingly a greater need for tests or examinations to permit of identification as to process as well as for content and quality. Such tests may consist of a metallographic study of the cross-section. Heretofore microscopic studies have been utilized chiefly in the examination of watchcase and optical goods, the great bulk of which are composed of "duplex" metals or gold-clad base metal.

Metallographic examination permits, in addition, the measurement of thickness of the gold-alloy layer. The specification for gold-filled watchcases according to the code referred to, so marked and sold in interstate commerce, calls for a minimum thickness of 0.003 in. for the outside gold-alloy layer and 0.001 in. for the inner. The requirements for rolled-gold plate cases, however, call for 0.0015 in. on the back but make no other restrictions. It must be recognized that drawing and spinning operations may and usually do change the thickness of the gold layer in different points. This may result in a considerable thickening or thinning of the gold layer. So too do pickling and polishing.

The microscope is also necessary in studying uniformity and proper distribution of the layer, inlay or shell of gold alloy. Again it offers a ready means of disclosing pits and imperfections, particularly in electroplate and soldered joints. The nature and compactness of the cable end of an optical temple, for example, is also best examined under suitable magnification for the tightness of wrapping and consequent resistance to the penetration of perspiration. It also may permit the measurement of thickness of the rhodium electroplate often applied as a finish or coloring to white-gold articles. Again, the microscope will be of especial value in examinations of articles made from compressed metal powders or case-hardened as described in a recent patent.

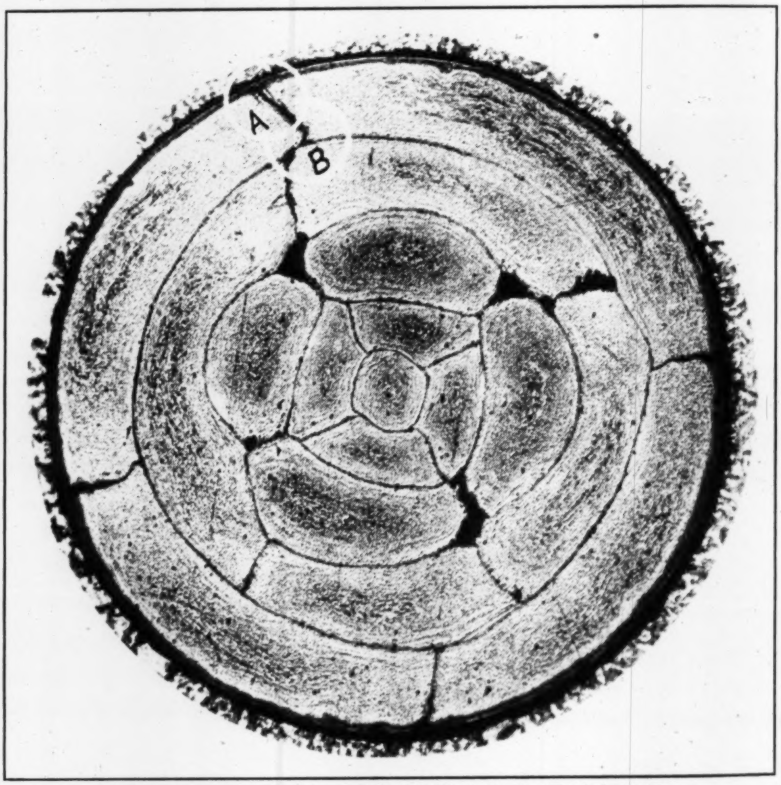

FIG. 2.—Cross-Section of Gold Clad Cable End (× 50).

Figure 2 shows the cross-section of an optical cable as etched with ammonium persulfate and potassium cyanide. The area in circle A shows the outer layer of gold alloy at junction of two strands of wire while that in circle B shows the junction of two outer and two inner strands. The canal is disclosed as extending to the fourth layer thus affording a medium for penetration of perspiration and consequent corrosion were it not for the protecting envelope of electroplated gold alloy at the periphery. The outside crystalline ring is a portion of the mounting alloy.

Physical Tests:

The demand for physical tests is comparatively limited on articles of adornment such as jewelry. In those pieces more utilitarian in nature, the matter of strength is more vital, particularly in optical frames. Here the bending and fitting of a bridge, pad-arm or temple, subject the metal to considerable strains. Fatigue and springiness are factors. While there are other notable exceptions, in general effort is made to use an alloy which will not work-harden excessively, but hardness testers find their present use almost exclusively in the die or tool room. Of course, material which must be heavily worked must have sufficient inherent or attainable physical properties to stand the sometimes drastic punishment to which it is subjected during fabrication. In addition the increased price of gold will tend towards lighter articles which in turn will call for increased stiffness and rigidity.

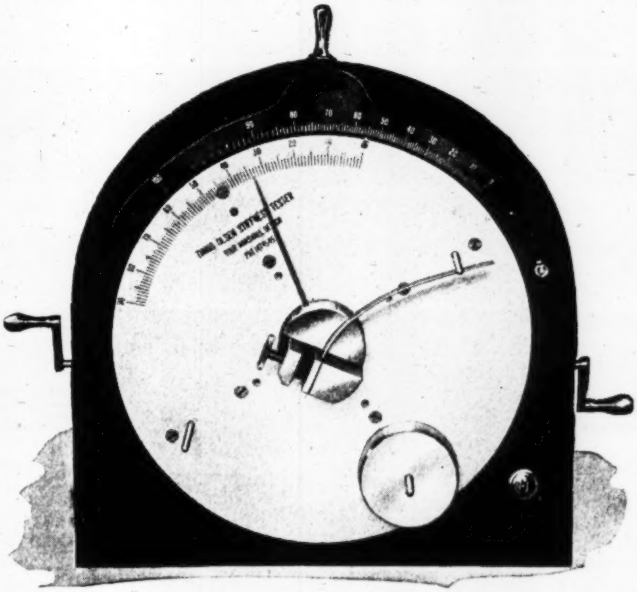

FIG. 3.—Tour-Marshall Stiffness Tester.

The Tour-Marshall Stiffness Tester, Fig. 3., developed in our laboratories is a compact machine working on a simple cantilever beam principle to give direct readings of the stiffness of wires, ribbon, flat strip, or simple fabricated shapes. It is adapted to materials from 0.01 to 0.1 in. in diameter or thickness and widths up to 1 in. It serves not only to check incoming material and control production but also to aid in research and development work. Tests for comparative values or conformity to specifications take but a few moments each.

Stripping:

While most tests for gold in alloys are carried out in the assay laboratory and furnace methods play a major part, nevertheless it is often more convenient, as in testing for "quality," to precede the "fire" portion of the assay by a stripping operation. Gold-clad metals may consist of:

1. A base of composition metal, as it is known in the jewelry or watch-case industry.
2. A similar alloy known as optical metal or O.M.
3. Nickel silver (5 to 30 per cent nickel).
4. Pure nickel.
5. Monel metal.
6. Stainless steel.

The higher nickel alloys and the stainless steel are of comparatively recent introduction and give rise to difficulties in assaying which if uncorrected tend to result in low assays. These metals do not, however, influence the true recoveries by the smelter. It is good practice to remove as much as possible of the base before subjecting the metal to scorification and cupellation. This is done by digesting the duplex alloy with a suitable solvent. Since certain of the metals used as a base or core are corrosion resistant, the removal by solution without appreciably affecting the inlay or outer gold shell is obviously difficult. Stripping may tend towards either a raising or a lowering of the karat unless properly carried out.

On occasion, it is absolutely necessary to ascertain the actual karat[1] although it can often be approximated closely enough for many purposes by calculation. Such calculations, however, may be misleading particularly on plate clad on both sides, for double plate, as it is known, may be composed, for example, of 1/10–14 K alloy on one side and 1/20–10 K on the other and a given ratio by weight of one karat will, of course, calculate to a different ratio of another: thus 1/10–12 K single plate = 1/20–12 K double plate. This would, of course, also calculate out to 1/15–18 K but probability would indicate otherwise since that is not a usual combination.

The stripping operation may also be complicated by the varying nobility of the inlay for this is not altogether a matter of the gold fineness. In the low-karat alloys in particular, formula is important also. While no minimum karat is specified by the National Stamping Act(3), the commercial standard and code previously referred to specifies 10 K, although jewelry manufactured for export may be 9 or 8 K or even lower. Fortunately, the most commonly used corrosion-resistant bases or cores are soluble in nitric acid. The greatest possible dilution is used in combination with the lowest convenient temperature as in stripping white gold on nickel or nickel silver. Greater care must be exercised when stripping "pink" or "coral" gold.

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[1] 1 karat (K) = 100 ÷ 24 = 4.167 per cent = 41.67 Fine = 41.67 parts per thousand or = 0.04167.

The several shades of yellow gold, being plated on high copper alloys as a rule, may usually be stripped with the alkaline solution developed some years ago by the author, or as modified by Gilcrist at the National Bureau of Standards(8).

The problem of stripping from stainless steel is not now important as this base is confined largely to stock in which the ratio is so extremely low that a regular assay for gold content will suffice.

Quite obviously, wire gives the same problems as double plate, since the base, except for the ends, is completely encased. Cutting the samples into small pieces of course increases the surface for acid attack. It decreases time and temperature but not necessarily acid concentration.

Assaying:

In determining the gold content of plate or wire several major assaying problems present themselves. One deals with trade-practice, one with the price industry can afford for the test, one with the obtaining of a representative sample. Their consideration is fundamental to a proper appreciation of the problem of testing.

It will be seen that tests in the precious-metal industries enumerated here are essentially tests of "value" and of "quality." For gold is a valuable metal and the assayer measures in hundredths or thousandths of milligrams where the chemist measures in tenths.

Yet assay methods do not always give true figures any more than do many chemical methods, despite the too general impression to the contrary. "Quality" and "value" are not always synonomous terms. "Quality" is the term used in reference to the gold content of a finished article or perhaps of the semi-fabricated material such as gold plate. The assay methods used, so far as the furnace and subsequent procedure is concerned, are similar to those employed on the skeleton scrap and other waste metal known variously according to the source as platers, optical, pencil, or watchcase scrap as distinguished from karat gold or dental gold scrap. Karat gold, of course, will show high gold values and dental scrap will usually carry platinum and palladium as well.

The so-called "commercial assay" methods on material of 100 fineness or less (and substantially all rolled-gold plate is that) do not give the full gold, unless by compensating errors. The variation from the true could be determined for a particular combination of metals, of course, but since usually the formula of neither inlay nor base alloy is known some compromise must be reached. The compromise no doubt often falls short of the ideal. Furthermore the manufacturer by act and by trade agreement is allowed a tolerance for the gold alloy *per se* of $\frac{1}{2}$ to 1 karat (20.83 to 41.67 parts per thousand). Advantage of this has naturally been taken by the assayer, pressed as he too often is for rapid and inexpensive assays. A tolerance of 10 per cent is allowed on gold-filled and rolled-gold plate.

Since comparatively few manufacturers have assay departments of their own, the assays for "quality" have in large part been performed (often as a courtesy or sales service at little or no direct remuneration) by those concerned chiefly in making the assays for "value." In other words, what is known as a "commercial assay" as against a "corrected" or "proof" assay is the rule. Corrected assays when used are usually confined to high-grade material such as solid. The correction, which may be either a plus or minus one, is arrived at by reassaying the slags and cupels from the "commercial assay." Any gold (or silver) so recovered is added to the original or main assay figure.

The proof assay is different. This presupposes a preliminary examination (if no data are available) for approximate composition. A synthetic alloy is then made up and run concurrently with the sample being assayed for "value" or "quality." Proof assays are standard practice in the valuation of refined gold and silver and in rich intermediate products such as Dore bullion. In theory all solid gold assays for either value or quality should be so run. In practice an assayer handling a certain type of material will attempt to develop an assay procedure in which compensating errors can occur. It must be apparent, therefore, that the proof assay gives figures of high precision. It is not so apparent that the method of slag and cupel correction does not give, necessarily, results of as great precision although the final figure so obtained may be quite accurate. Here again compensating errors may help. While a treatment of the slag and cupels may not result in full recovery of the gold (and/or silver) yet the gold bead as parted will always contain silver and if the article had been rhodium plated will certainly carry rhodium. This lack of full recovery may be more than compensated for by these retentions in the bead, thus giving a high result, but the "commercial assay" of common usage for "content" or "value" is on the whole one of low tendency(11).

"Value" assays are those made in connection with the disposal of scrap and sweeps to smelters and refiners. The extent to which such assays may be low is a variable, depending in large part on the nature of the base. In platers scrap the higher the nickel or tin the more appreciable the loss, particularly if silver is not present in proper ratio. At least 5 parts silver to 1 part gold is used in our laboratories.

Coupled with a recognition of a generally low tendency with high nickel and tin has been that of the difficulty of obtaining reproducible results. Given synthetic alloys or properly prepared samples of platers scrap—homogeneous medium size moss or shot—there is no difficulty in getting check assays from the *same furnace* run. Seemingly duplicate sets, run at another time or in another furnace, may give results as much as 0.10 to 0.25 fineness apart from the original figures. The answer is in the control of temperature and oxidation.

The buying and selling of platers scrap is conducted, however, under fairly consistent trade practices. While the contract may call for payment for "full" gold contents less the refining and smelting charges, yet it has been generally accepted that the "commercial" assay method would be used. It is evident then that it is incumbent upon the assayers to employ a method or methods which will give the greatest precision short of running slag and cupel corrections or proofs. Assay fees are one limiting factor; speed is another. Knowing the approximate or expected error for that laboratory the ideal "commercial method" may suffice for assay of "value," provided the sample is representative and homogeneous, or contrariwise it may suffice for a "quality" assay because the integrity of the sample may not be all that is desired.

Sampling:

Sampling then becomes of major importance to the assayer as well as to his client, for the amount to be weighed as a charge is part and parcel of the method. The number of checks or determinations depends on the method of sampling the platers scrap and the method of choosing and the number and weight of sections chosen of the rolled-gold plate or filled stock, or again the finished optical frame, bracelet or watchcase. In the latter cases often only one specimen is available.

Sampling scrap is one thing. Sampling duplex metal is another. Sampling a finished product is still another. Scrap may be sampled in lots weighing up to 13,000 troy ounces, the maximum capacity of the usual crucible, for all larger lots are best melted and cast into bars or anodes. If the weight is small the resultant bar may be sampled by drilling, preferably using a 6-hole diagonal template and a small drill.

For best results a shot sample is taken, just prior to pouring or ladling into the molds, by dipping either a hot ladle or clay crucible into the molten metal. The melt should be first thoroughly mixed by stirring and the dross or slag skimmed off and reserved for the time being. Then the sampling ladle is dipped into the melt and the contents poured slowly from a suitable height into a clean pail of water either with constant stirring to break up the stream or the molten metal may be comminuted by impinging on a slanting board at a point just sufficiently below the surface of the water to give a minimum of very fine or very coarse shot.

A mossy type is preferred to round compact shot. When properly taken the very coarse and very fine portions of the sample may be screened out—the coarse because it is inconvenient for the assayer to cut and handle at the balance, the fines because it tends to become oxidized during passage through the air and thus gives inordinately low results. The same low result may occur due to contamination with such dross or slag from the surface of the melt as may have formed during skimming or may not have been thoroughly removed. Such slag tends to break up into a fine powder on striking the water, or may become entangled in the mossy shot if poured from too great a height.

TABLE I.—SEGREGATION EXPERIMENT IN MOLTEN PLATERS SCRAP.
Balance—Copper, Zinc, Nickel, Silver and impurities in the order named.

	Gold Only Assayed, parts per thousand[a]			
	Lot No. 58/C7404	Lot No. 59/C7415	Lot No. 833/C7411	Lot No. 841/C7419
Regular sample, top quarter of melt (*R*)	124.04	102.96	24.98	23.60
Special sample, $\frac{1}{4}$ poured off (*A*)	124.08	102.81	24.71	23.49
Special sample, $\frac{1}{2}$ poured off (*B*)	123.60	103.13	24.89	23.50
Special sample, $\frac{3}{4}$ poured off (*C*)	124.03	102.91	24.87	23.59
Special sample, composite of top and $\frac{1}{2}$ (*D*)	123.99	102.92	24.84	23.55
Special sample, composite of $\frac{1}{4}$ and $\frac{3}{4}$ (*E*)	123.95	102.77	24.82	23.57
Calculated average of regular and $\frac{1}{2}$ (*R* and *B*)	123.82	103.05	24.93	23.55
Calculated average of $\frac{1}{4}$ and $\frac{3}{4}$ (*A* and *C*)	124.05	102.86	24.79	23.54
Calculated average of Regular $+ \frac{1}{4} + \frac{1}{2} + \frac{3}{4}$	123.94	102.95	24.86	23.55
Calculated average of *D* and *E*	123.97	102.85	24.83	23.56
Calculated grand average	123.95	102.92	24.85	23.55

[a] Results shown for *R*, *A*, *B*, *C*, *D* and *E* represent an average of two or more determinations.

Obviously the shipment must have been weighed before melting to check the shipper's weights and must be weighed again to obtain the after-melting weight to which the assay applies. The after-melting weight is always less due to loss of zinc as fumes or dross during melting. The skimmings, as reserved, are recharged into the pot—together with some charcoal and the heat raised. After sweating the reduced metal is poured into water and the charcoal remaining having been floated off, the recovered metal from the now clean crucible or pot is dried and the weight added to that of the main bulk.

If stirring has been thorough, the sample may be dipped for convenience from just below the surface. That this is so will be seen from some experimental data given in Table I. These results are each representative of least duplicate assays for any one of the samples. As added security the experiments were carried out on two lots of high-grade watchcase scrap and two lots of medium low-grade platers scrap. The results not only are

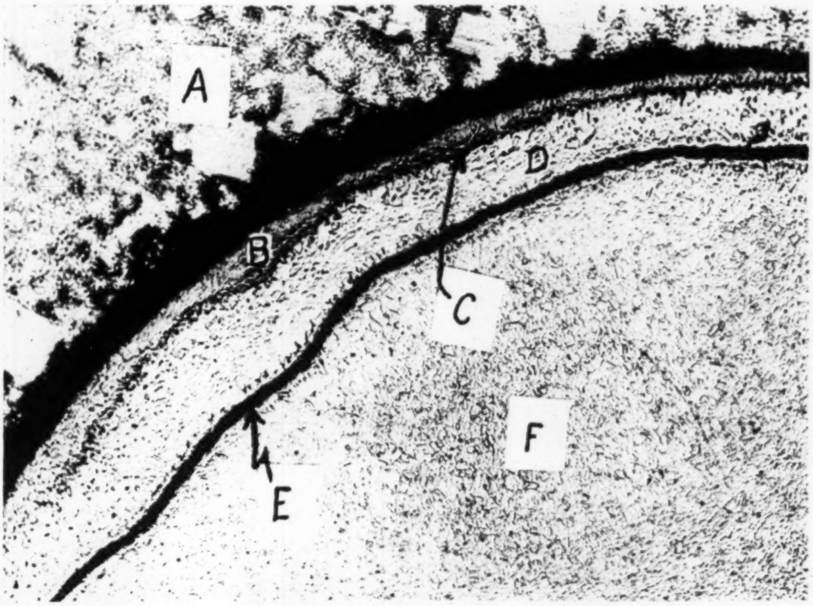

FIG. 4.—Pink Gold-Clad Wire—Swaged and Drawn (× 100).

A	Mounting alloy	*D*	Base alloy bronze or optical metal
B	Pink gold alloy or shell	*E*	Solder diffusion zone
C	Solder diffusion zone	*F*	Optical metal core of wire

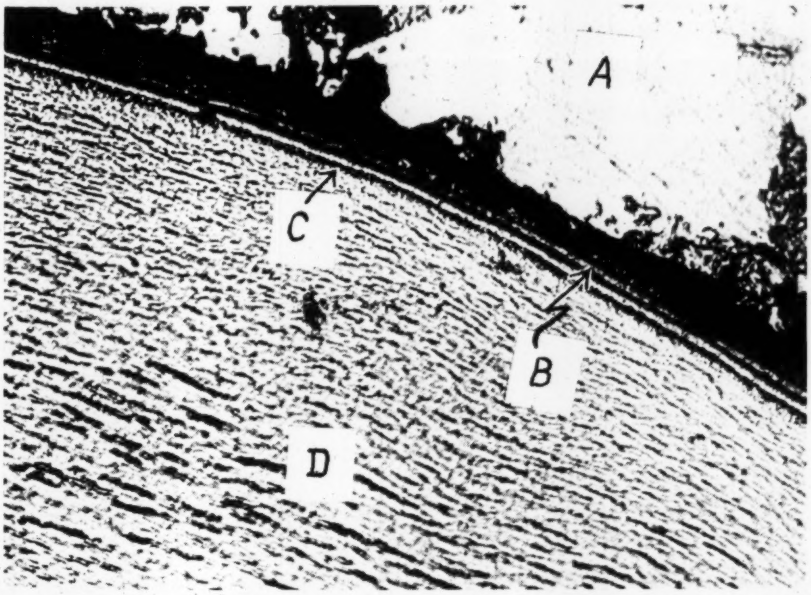

FIG. 5.—Pink Gold-Clad Wire Electrodeposited Alloy After Heat Treatment (× 500).

A	Mounting alloy	*C*	Diffusion zone
B	Electrodeposited alloy	*D*	Base alloy core

representative of various strata of the molten charge and, therefore, of the shipment (see *R*, *A*, *B* and *C*), but also show the homogeneousness of the sample, for in *D* and *E* the assay charge was weighed out at the balance after compositing equal amounts of *R* plus *B* and *A* plus *C*.

Each lot weighed approximately 12,000 oz. and the same method of assay for gold was employed. Silver being low and negligible in value as compared to gold and the gold assay method used not being applicable, it was not determined but it was assumed that the same deduction as to uniformity could be made. If a slag and cupel correction had been run such minor discrepancies as do appear would likely have been smoothed out.

Sampling rolled-gold plate or wire, however, requires an altogether different technique and is in many ways more difficult. Due to variations in hardness of the gold alloy and of the base and to a certain extent to variation in initial thickness from point to point, the gold value also varies. Generally speaking, the colored gold alloys are softer than the base or core. The method of fabrication also has an influence, swaged wire for example tending to vary more than rolled wire as was shown some years ago in a graph submitted by the author as discussion of an excellent paper by E. A. Smith(9).

The method of cutting the sample is, therefore, extremely important, even allowing that due to the high cost of the material and other reasons the samples submitted to the commercial laboratory are often insufficient and perhaps not as representative as they should be. But whether the sample is to be employed for assay of the gold alloy layer or the over-all assay on the one hand or for microscopic studies, every care should be taken to avoid a "smearing" of the softer alloy on the hard. This almost invariably happens when shears are used and usually will cause erroneous results as the gold alloy being softer tends to be drawn down by the cutting knife over the base metal core or layer. As a result an assay for quality will tend to be too high. For the same reason, in measurements of thickness—particularly important in watchcase plate and in other studies of distribution for wear or corrosion resistance—the result on the softer layer will be too high.

Quality Tests on Plate:

Emphasis being laid more and more today on quality, the manufacturer, even more than the consumer, is studying distribution and also the actual karat of the gold layer.

The trade-practice standards referred to previously and certain requirements of the U. S. Customs have also resulted in greater recourse to both the microscope and stripping operations. The microscope serves best to distinguish between material made by the rolled-gold process (see Fig. 4) and that made by either the ordinary or heat-treated electroplate (see Fig. 5).

Figure 4 represents a transverse section of pink gold clad wire as swaged and at 100 diameters. The etchant was ammonium persulfate and potassium cyanide. *A* is the matrix alloy. The dark unmarked area is the void often present. This sample was not plated with copper as a protection during preparation. *B* is the shell of pink gold alloy and *C* is a solder diffusion zone bonding *B* to *D*, a layer of optical metal. *E* is a solder-diffusion zone showing penetration into the layers *D* and *F* of optical metal. Study of this section not only disclosed first that the duplex wire was made by a mechanical process and second the efficiency of the bond and somewhat of its nature but it also showed definitely that the wire was made in two operations. That is, a shell or tube of gold-filled stock was in turn soldered to a core of base metal. Figure 5, on the other hand, represents a transverse section of pink gold clad wire made by electrodeposition. This specimen was etched as before but the magnification is at 500 diameters. *A* again represents the mounting alloy and the adjacent dark area the apparent void, *B* is the electrodeposited alloy after heat treatment. The stratified nature of the deposit is apparent. *C* has reference to the diffusion zone and *D* is the base metal core.

Stripping is of particular service in the higher karat or more noble gold alloys plated on the more readily dissolved base metal or alloy. As the gold layer in rolled gold is bonded to the base by first interposing a thin strip of suitable solder and then subjecting to heat treatment, there results an intermediate diffusion zone of varying nobility and consequent resistance to acid attack. While there are obvious objections to the method they are not as real as they appear and an assay of the gold layer usually furnishes the additional data desired. Welded rolled gold plate is now made.

The most common test is the old one of testing the resistance of plate by nitric acid either by spotting or by the use of the touchstone. The first is used to quickly test whether the gold alloy layer is sufficient or the karat too low. The touchstone test in effect consists of comparing the streak made by rubbing the sample on a piece of black slatestone with the streak of standard pencils of 10, 12, 14 karat, etc., alloys. The streaks having been made under like conditions and adjacent, a drop of fresh aqua regia is applied to each. The change of color and rate of disappearance give the necessary qualitative-quantitative data desired.

Samples for stripping should be weighed carefully before subjecting to either acid or alkaline attack. On low-grade material the layer of gold is often but a film and filtering must be resorted to. The great bulk of assays, however, are for "over-all" quality and cutting errors are all-important. They may be minimized by careful use of a fine-tooth hacksaw applied strictly perpendicular to the longitudinal axis. If the samples have been cut by shears, the ends should be trued off by grinding on fine emery. Too heavy a pressure should be avoided to prevent a buffing or smearing action.

Articles of the jewelry and optical type wherever possible should be assayed as received except for the removal of those parts legally exempt. In cases of possible civil or criminal suits it is sometimes necessary to reserve one-half (preferably the half stamped with the quality mark). As many articles are assembled, the parts may represent as many different heats of metal and even more than one manufacturer. More important still, the article has in many cases been soldered one or more times, thus employing solders of varying melting point and perhaps different gold content. Great care should accordingly be taken that the portion reserved is in effect a substantial duplicate of that used for assay.

It will be apparent, even with the limited examples given, that obtaining a fair and representative gold figure is not a simple matter. It is probably mainly for this reason that no concerted effort has been made to arrive at a standard procedure for the assay itself. Except in the case of scrap sold to the refinery, very few samples are ever subjected to assay by two assayers. If the results, as obtained in the two laboratories, checked they have been generally assumed to be correct. Inasmuch as the rolled gold plater has had still another bookkeeping check, by adding the gold content of his scrap to the calculated gold content of the stock as sold and comparing with the refined gold purchased, it will be seen that assays of scrap at least must have been sufficiently close to the truth. The use of high amounts of nickel and the new standards for rolled-gold plate both call for study and comparison of present assay methods and the respective errors.

TABLE II.—MATTE SAMPLE.
Gold in parts per thousand, 1 g. taken.

Lot	Laboratory A	Laboratory B	Laboratory C	Range	Average
No. 2A	37.06	37.10	37.23	0.17	37.13
No. 3	24.62	24.70	24.33	0.37	24.55
No. 4	31.06	31.07	30.93	0.14	31.02

TABLE III.—SHOTTED SAMPLE.
Gold in parts per thousand, 1 g. taken.

Lot	Laboratory A	Laboratory B	Laboratory C	Range	Average
No. 1	96.74	96.70	96.47	0.27	96.64
No. 2	33.83	33.90	33.88	0.07	33.87
No. 1A	73.07	73.07	73.06	0.01	73.07
No. 3A	39.28	39.30	39.20	0.10	39.26

This point is best illustrated in connection with some experimental work where three assayers were involved. The material was scrap originally carrying 12 K gold plate on pure nickel but sampled in the form of a powdered matte.

When the high-nickel scrap first began to appear at the refineries it was necessary to devise a new method of obtaining the sample due to inability to get high enough temperatures in the coke, gas or oil furnaces available. We at first suggested melting with sulfur, pouring and pulverizing the resultant matte as above. While this gave a very homogeneous sample and lowered the nickel content the sulfur fumes made the operation a very disagreeable one. Later we suggested first melting approximately an equivalent weight of copper and then adding the high-nickel scrap until melted. This reduced the percentage of nickel in the sample and the melting point of the charge and is the method now specified by us.

Table II represents the material above while Table III gives results by three different assayers on a sample of a melted mixture of 12 K plated partly on 15 per cent nickel silver and partly on composition or optical metal. Each figure in itself is the average of two to six runs, the variations in any one laboratory having been as great as 0.40 parts per thousand.

As the results in any one laboratory on a shotted sample should check within 10 parts per thousand and between two laboratories the maximum allowance should in no event be more than 0.15 parts per thousand, it was decided to conduct a series of experiments that it was hoped would result in an improved method less productive of anomalous results.

The results given in both Tables II and III were presumably all obtained by scorifying once, cupelling, and parting. The number of grams of lead used in all cases is not known. Our own experience had shown that it was imperative that excess lead should be present and 90 g. is used. Scorification should be carried through at a temperature not less than 1100 C. Lesser amounts of lead and lower temperatures may give a higher melting, more viscous slag more retentive of shot and, therefore, more erratic in result. The results tended to be less than the true figure by about 0.15 parts per thousand as checked by direct recovery of the gold using an all-analytical method—tedious and expensive but sure—and by employing synthetic alloy charges.

SUMMARY

An attempt has been made to lay the foundation for the standardization of tests of gold-clad material. The survey covers a number of the salient points but being general in character omits, of necessity, many important details. The advantages and fields of the several methods of testing have been considered as have been the limitations of speed, of cost and the small weights of samples often available.

The field is one covering a myriad variety of articles. Design, color and appearance are often of paramount importance, yet the distinctive feature is the precious metal. Gold, for example, has a high intrinsic and sentimental, yet withal, a high technical value, because of corrosion resistance and relatively easy working qualities.

Some standards of quality as mentioned having been set, it would probably be in order for the Society to establish standard methods of testing. It is believed that all responsible members of this important industry would welcome a standardization of the basic methods of testing certain of their products. Later on, perhaps, the Society might cooperate in setting up standards of quality. This paper is presented as a contribution to that end.

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DISCUSSION

MR. R. H. LEACH[1] (*presented in written form*).—We are indebted to Mr. Wright not only for giving us such a comprehensive summary of the more important uses of precious metals, but also for indicating how this Society could be of assistance to both producers and consumers of these metals.

The second part of the paper is of particular interest because of the complete manner in which the problems involved in establishing standard specifications, methods of sampling, and methods of testing for duplex plate are discussed. Although Mr. Wright has selected gold-clad metals, the same problems are involved in developing standards for duplex metals produced from other precious metals or their alloys.

It is hoped that this paper will serve to bring to the attention of the users of precious metals, the facilities that the Society offers whereby both producers and consumers may cooperate in the development of standards which should be as helpful in the precious metal field as they have been in other fields.

MR. EDWARD WICHERS[2] (*presented in written form*).—Mr. Wright should be commended for thrusting the plow into the almost unbroken, stubborn soil of this "field" of standardization. As he clearly indicates, there will have to be much clod-breaking, stone gathering, harrowing, and whatever other forms of ground-preparing one can think of, before the field will produce a suitable crop of methods and standards.

To forsake agricultural metaphors, two matters which the author has stressed may be noted for the sake of further emphasis. One is the importance and difficulty of proper sampling. The other is that the precious metals are finding increasing usefulness in ways which depend upon certain desirable chemical and physical properties rather than upon their costliness. Even in popular jewelry there is a large demand for articles which have a maximum of decorative value with a minimum of intrinsic value. As shown in the paper, methods of assaying or chemical analysis for purposes of evaluation (except for complicated platinum metal mixtures and alloys) are reasonably adequate. Methods for determining resistance to wear and corrosion are in their beginnings, and, so far as known, the all-important correlation of such tests with behavior of materials in service is still in the future.

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[1] Manager of Research and Development, Handy & Harman, Bridgeport, Conn.

[2] Chemist, National Bureau of Standards, Washington, D. C.

The following comments on details are offered to clarify or to supplement the author's treatment of them:

With reference to contact points, silver finds much use, but not in automobiles, where tungsten is used almost exclusively. Platinum alloys are believed still to be used in magnetos for aviation engines.

While methods for determining the weight or thickness of silver in silver-plated articles may not be in general use, they are included in Federal Specification RR-T-51a for silver plated tableware.

One might question whether the thickness of rhodium electroplate is sufficient to be determined by measurement under the microscope. The limit of such measurements is probably above 0.00001 in. whereas the thickness of the plated coatings is usually less than that.

The term "quality" is used with two meanings, but the reader need only note that it is printed in quotation marks when used in connection with an assay for gold or silver content. Perhaps confusion could be further avoided by using the term "serviceability tests" instead of "quality tests" in connection with duplex alloys.

MR. J. R. TOWNSEND.[1]—We have had occasion to make comparative corrosion tests on a great variety of base metals and precious metals in connection with the development of electrical contacts for telephone use. In carrying out comparative tests, we used, in one test, a corrosion method consisting of an atmosphere one thousand times more concentrated than the worst industrial atmosphere that we were able to find in the country. This consisted of 5 per cent carbon dioxide, 0.5 per cent sulfur dioxide, 0.5 per cent hydrogen sulfide and 85 per cent relative humidity. After exposures of from three to six months, we were able to develop tarnish films on a great many of these metals. However, if with the exposure of these metals to this atmosphere, we combine intermittent wear, say several rotations of a disk against a small contact spring about $\frac{1}{8}$ in. wide and 0.020 in. thick and a pressure of about 20 g., we find that the corrosion of tarnish film in every case is greatly accelerated; in fact, gold will not develop a noticeable tarnish film when exposed to this atmosphere, but when rubbed in this way will develop a tarnish film. All of the common base metal alloys, stainless steel, and silver, gold and platinum base alloys have been tried, and in every case with abrasion or rubbing the tarnish film was accelerated. This is probably due to the fact that the metal in the rubbed area was strained by the rubbing and produced a condition where it was cathodic to the surrounding metal and more corrosion took place. It is concluded therefore that in any tarnish or corrosion test on precious metals, the question of rubbing or abrasion is extremely important and should be considered. One or two points of interest in this test was that stainless steel of the 18 per cent chromium, 8 per cent nickel type showed no tarnish film or discoloration, whereas the gold did tarnish and showed discoloration under the test.

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[1] General Development Laboratory, Bell Telephone Laboratories, Inc., New York City.

MISS C. M. HOKE[1] (*by letter*).—We are glad to see that Mr. Wright has not neglected to mention the wide usefulness of the approximate and "commercial" assays, which are employed in cases where assay fees must be kept low or where time is a major factor. In our opinion, these tests, when their import is properly understood, are of great value to the industry, and any laboratory that fails to make them is losing an opportunity to be of genuine service to this field.

To explain: A jeweler has a button, or a jar of solution, or a box of scrap, the whole value of which may be at most $50 or so; he wants to know its approximate value, but plainly the job does not justify a very precise nor complete assay, especially as platinum, iridium, and palladium may be present in addition to gold and silver. Yet if he could obtain at a low fee, within a few hours, an approximation that might do little more than tell him whether to throw the stuff away or try to sell it, he would be happy and the laboratory would have a piece of work which might lead to other and more ambitious undertakings. When it is recalled that even a small jewelry shop might have dozens of such jobs in the course of a year, it would seem that here is a field for endeavor.

Mr. Wright mentions the touchstone test as applied to plate. It is of course used even more frequently upon "solid" (karat) gold. Within the last few months hundreds of thousands of gold articles have changed hands on the basis of touchstone tests alone without benefit of assay, the value involved being millions of dollars. Both buyers and sellers knew that the touchstone test was only an approximation, yet (because of the expense and delay of a complete assay) they were willing to accept these approximations.

In our opinion, the precious metal industry has real need for the several tests described so clearly by Mr. Wright, and also (if they can be worked out) for a series of approximate tests to cover buttons, alloys, solutions, and also articles such as jewelry whose form should not be noticeably damaged. Presumably these tests would be based on preliminary work characterized by high accuracy. As to their nature I do not presume to make suggestions.

MR. A. W. PETERSON[2] (*by letter*).—Referring to the testing requirements for duplex metal products, Mr. Wright mentions some interesting uses of the microscope, but we feel that it could also be used to advantage in determining very closely the quality of plate in a simple and rapid manner. Reference is made in the paper to the difficulty in calculating the exact karat in the case of double plate and examples are given (page 352) of how various qualities and karats may be calculated into terms of different karats and qualities. By simply examining a cross-section of the metal first under the microscope, the quality may be quite definitely determined. We use this simple scheme very successfully as a checkup device.

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[1] Consulting Chemist, Jewelers Technical Advice Co., New York City.
[2] Metallurgist, General Plate Co., Attleboro, Mass.

On page 352 it is stated: "The stripping operation may also be complicated by the varying nobility of the inlay for this is not altogether a matter of the gold fineness." We wonder whether the reference here is to the base metals which may be used in the gold alloy—nickel against copper, for example. We found that in general, especially of the 10-karat alloys and better, that the nobility of the alloy depends upon the atomic percentage of noble metal present.

Regarding commercial assaying practice we agree that the proof assay is the ideal but for the cost involved. Perhaps the so-called "compensating errors" are justifiable in the case of scrap assays as they are the only economical means of assaying small lots of scrap. However, we believe it would pay to run proofs on large lots of miscellaneous scrap. But, allowing for "compensating errors" may be a bit dangerous. What if no silver or rhodium are present? Even when silver is present it should be removed if parting is complete, as it should be when carefully carried out.

We believe that it would be wise to segregate scrap according to base metal types and determine the variation from the true for these combinations. If the manufacturer would do this, especially for lots of great quantity, he could expect greater returns. However, it is a question whether numerous assays on segregated lots of scrap of this nature would be more costly than the losses sustained by the assays being low on a bulk lot of scrap having an unknown type of base metal. It should be advantageous for the customer to state the type of base metal, if possible, when desiring an assay of gold plate as it would result in a better assay.

In any case, we believe the corrections for scorification and cupel losses should be applied. We see no reason why duplicate results should not check more closely than the results given even when run in a different furnace. We have devices today whereby furnace temperatures and atmospheres are easily kept constant; these could very easily be applied to the assay furnace as well.

The example illustration of how the microscope may be used to distinguish between rolled-gold plate and heat-treated electroplate would have been still more convincing if the author had also included a specimen of welded rolled-gold plate. It is often very difficult to distinguish between a rolled-gold plate in which the gold sheet has been bonded or welded directly, using no solder, to the base metal and a well diffused electroplate. Three such samples would give a complete picture.

MR. E. A. CAPILLON[1] (*by letter*).—Mr. Wright mentions the difficulties in refining and assaying which were brought about by the introduction of rolled-gold plate on high nickel bases such as monel metal and pure nickel. Our experience with rolled-gold plate on pure nickel has shown that despite statements to the contrary by some refineries there still exists a large gold loss in the refining of such scrap. On numerous occasions we have plated test bars with definite amounts of karat gold and kept careful check of weights after these bars were soldered and cold rolled. As will be seen from the figures given in the accompanying table the losses incurred in soldering and rolling the stock in no way account for the excessive gold losses as reported by the refineries:

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[1] Metallurgist, The D. E. Makepeace Co., Attleboro, Mass.

	TEST 1	TEST 2	TEST 3	TEST 4
(a) Weight of 12 K white gold	146.78	131.70	129.70	112.48
(b) Weight of pure nickel base	1236.90	1057.30	1026.00	1060.00
(c) Weight of silver solder	8.04	7.20	7.10	7.20
(d) Total a, b and c	1391.72	1196.20	1162.80	1179.68
(e) Weight of bar after soldering	1391.60	1194.90	1162.60	1177.46
(f) Loss in soldering (d minus e)	0.12	1.30	0.20	2.22
(g) Weight of bar after cold rolling	1390.74	1194.50	1161.70	1177.00
(h) Loss in rolling (e minus g)	0.86	0.40	0.90	0.46
(i) Weight 12 K to refinery (a minus f)	146.66	130.40	129.50	110.26
(j) Average assay 12 K gold	0.5009	0.4955	0.5001	0.4986
(k) Weight fine gold to refinery (i multiplied by j)	73.46	64.61	64.76	54.98
(l) Fine gold reported by refinery	70.60	61.20	59.80	53.33
(m) Weight gold lost (k minus l)	2.86	3.41	4.96	1.65
(n) Gold lost, per cent	3.89	5.28	7.65	3.00

Weight figures are in pennyweights Troy.

NOTES.—It is assumed that the soldering loss is 12 K gold and therefore this loss is subtracted from (a) to obtain item (i), the weight of 12 K gold sent to refinery. The soldering loss is caused by the molten solder flushing some gold away in drops which fall to the bottom of the soldering furnace. Actually the gold content of these drops is considerably less than 50 per cent due to dilution by the solder.

The loss in weight in rolling (item h) is neglected in calculating gold to refinery (item i) as the composite soldered stock contains only about 5 per cent fine gold as compared to 50 per cent fine gold in the original 12 K gold. Moreover, the liberal allowance made for fine gold loss in soldering, as above, should more than compensate for this omission.

The results of three different refineries are represented in the above four tests.

The following facts are brought out:

1. In the four tests the lowest assay value of the 12 K gold is 0.4955, or less than 1 per cent from the desired 0.5000. When, however, these four 12 K white gold bars are plated on $\frac{3}{4}$ in. thick pure nickel bars and the composite bars cold-rolled to say 0.020 in. and refined to recover the fine gold, losses are incurred which run as high as 7.65 per cent shown in test No. 3.

2. That the presence of pure nickel is the cause of this excessive loss in gold is indicated by the fact that 12 K white gold contains some 15 per cent nickel while the rolled-gold plate made up as above contains over 90 per cent nickel.

3. Inasmuch as the tests represent results of three different refiners the losses cannot be attributed to faulty or slack methods of only one refiner.

4. The above figures indicate clearly that there is much room left for experiments in the assaying and refining of high nickel rolled-gold plate with a view of cutting down gold losses.

MR. F. C. MESLE[1] (*by letter*).—The paper deals largely with gold clad and plated metals, which seem to present more difficult and intricate problems for standardization quality and methods of testing than does silver-plated tableware in which I am particularly interested. The author calls attention to the need for definite quality standards, the terms in which these standards should be expressed, and the need for methods of measuring and checking. I am in complete agreement with him on these points as will be noted in my paper on "Standard Quality for Plated Tableware" published subsequently.[2] I have gone a little further and suggested what some of these standards should be, and a simplified method of measuring or checking these quality standards.

For many years I have been interested in the distribution of electroplated deposits and methods of control as well as methods of checking the distribution. An article by the writer dealing with some of these factors was printed in the American Electro-Platers' publication *Monthly Review* of November, 1928. This same article was printed in *Metal Industry* for March, 1929.

MR. THOMAS A. WRIGHT[3] (*author's closure by letter*).—I appreciate very much the helpful comments of the several discussers, certain features of which may perhaps bear further consideration or emphasis.

I am glad that Mr. Wichers has brought out that Federal Specification RR-T-51a includes a method of testing silver-plated tableware for weight and thickness of coating. In the paper, the reference to absence of methods of sample selection and determination was to the code provisions. Tungsten has replaced not only silver but platinum alloys in battery ignition contacts, although platinum-iridium of the 80–20 or 75–25 formula is used not only in air-craft magnetos but in automotive trucks fitted with dual-ignition.

It is true that 0.00001 in. is about the practical limit of actual microscopic thickness measurement and that the rhodium plating is often but a flash, yet these superficial coatings do show up at suitable magnifications. A statement to the effect that a coating is less than 0.00001 in. usually furnishes the answer required. Magnification at 500 diameters and above is often necessary.

From the viewpoint of the consumer, "serviceability tests" would often, but not always, be the proper term but from that of the National Stamping Act and that arising out of the relatively high material cost (of gold for example) "quality tests" would be more explicit. The two terms, therefore, are not strictly synonymous.

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[1] Oneida Ltd., Oneida, N. Y.

[2] F. C. Mesle, "Standard Quality for Plated Tableware," *Metal Industry* (New York), Vol. 33, No. 8, August, 1935, p. 283.

[3] Technical Director, Lucius Pitkin, Inc., New York City.

The comments made by Mr. Townsend are also informing. It must be remembered, however, that 0.5 per cent sulfur dioxide and 0.5 per cent hydrogen sulfide substantially cancel one another according to the equation:

$$2H_2S + SO_2 = 3\ S + 2H_2O.$$

This liberation (and precipitation) of free or elemental sulfur may well account for the comparative freedom from tarnishing except when a rubbing action is present.

Mr. Peterson's suggestion as to the use of the microscope in checking quality confirms our experience. That the nobility of the alloy is related to the atomic percentage of noble metal present confirms the work of Tamman and others abroad, but the reference in the paper was to the selective methods of attack used to strip the noble alloy from the underlying base. Nickel-base alloys are attacked less successfully than copper base for example.

Parting is rarely complete. The gold recovered is ordinarily only 998 fine at best due to silver retention. Rhodium is left with the gold and should at times be corrected for. Too much emphasis cannot be placed on Mr. Peterson's remarks about scrap. Proper assaying of these products calls for considerable knowledge of both the fabricating, refining and other metallurgical problems involved.

As to the data obtained by Mr. Capillon from the interesting tests described, it seems desirable to emphasize the necessity for very careful assaying to overcome the errors due to (1) sampling and preparation thereof, (2) multiplication of errors where small sample weights are used, (3) slag and cupel losses, (4) parting losses from floured gold, and (5) precision of weights employed. Only by carefully controlled research work involving synthetic alloys, temperature measurement and the recovery, weighing and assaying of every product or by-product can one arrive at a true conception of the magnitude of the error. Seven per cent tin can apparently cause assay errors as great as those due to say 90 per cent nickel. Properly carried out the slag loss in assaying synthetic alloys of those types containing accurately known amounts of gold equivalent to 25 fineness ($\frac{1}{20} - 12$ K) showed a maximum slag loss of 0.03 fine, a cupel loss not exceeding 0.18 and as low as 0.08 and a total unaccounted for fineness loss of 0.03. This indicates that a commercial assay by such a method would be low on the average about 0.15 fine, for established commercial practice calls for an *uncorrected* assay. There is reason for believing that quick, cheap assays may show losses far exceeding that. When, as is too often the case, the sample is open to question as to its representative character, the loss may even be greater or offset in whole or in part.

THE CHEMISTRY AND PHYSICS OF CONCRETE SHRINKAGE

By R. W. Carlson[1]

Synopsis

A simplified explanation is given of the manner in which the gel resulting from the hydration of portland cement causes shrinkage upon drying.

Attention is directed to the fact that all concretes shrink more than would be expected from the individual behaviors of cement paste and aggregate, and from the relative dimensions of each in the concrete. Although many possible explanations suggest themselves, it is indicated that voids around the aggregate particles and within the aggregate may be the most important factors causing concrete to shrink more than would be expected.

Data are presented showing the effect of size of mass and distance from an exposed surface upon the shrinkage of concrete. It is shown that in large masses the tendency to shrink is limited to the region within a few inches of the surface, even after 4 months of drying. Even a member of no more than one foot thickness may shrink but a fraction as much as a small bar during an average drying season.

The shrinkage of concrete upon drying is governed by the gelatinous constituents in the cement. Hydrated cement contains minutely porous calcium silicate gel the amount of which increases with the age of hydration and is greater for higher water-cement ratios and for finer cements. The amount of gel also depends upon the chemical composition of the cement; fully hydrated dicalcium silicate is mostly gel, while hydrated tricalcium silicate is somewhat more than half gel.

Besides the calcium silicate gel, cement contains other constituents which may contribute to shrinkage. Hydrated tricalcium aluminate gives up more and more water when exposed to drier air with consequent reduction in volume. This compound undoubtedly gives the greatest shrinkage per unit contained, but since it is not present in large amount in a standard cement, its total effect is not usually large.

The crystalline materials in cement are not believed to contribute to shrinkage. They are chiefly the unhydrated interiors of original grains and calcium hydroxide, a by-product of gelation. Within the ordinary range of humidities, these crystals do not change their volumes except as hydration continues. The quantity and characteristics of the calcium silicate gel, therefore, largely determine the potential shrinkage upon drying of hydrated cement.

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Moisture is held in the pores of the gel in hydrated cement by certain attractive forces which are large because the surface area within the pores is large in comparison with the volume of water held. In this sense, the water is in weak chemical combination with the gel. When water is removed from the pores by evaporation, the forces which previously attracted the water become effective in attracting the atoms of the gel closer together. Thus, the gel is mechanically or elastically compressed and its volume is reduced. For a given gel there is an equilibrium water content and corresponding volume for each atmospheric humidity to which it may be exposed. As the air becomes drier, the water content and the volume become smaller. In practice, the equilibrium water content may never be reached.

Time of exposure to drying and size of specimen are prime factors governing the actual shrinkage that will occur. The pores of calcium silicate gel are so small that for a given humidity or vapor pressure, full potential shrinkage is approached very slowly. In Table I are given measured values of contraction of small, neat cement bars ($1\frac{1}{2}$ by $1\frac{1}{2}$ by 12 in.) exposed on all sides to air of 50 per cent relative humidity. Note that although the most rapid shrinkage occurred during the early stages of drying, there is every indication that the contraction has not ceased at the age of 4 months. Bearing in mind that a maximum distance of only $\frac{3}{4}$ in. must be travelled by the moisture to reach the surface and escape, it becomes apparent that in field structures where larger dimensions are the rule, only a small part of the potential shrinkage may be realized during the course of the average drying season. Data on concrete specimens of appreciable size will be presented after first considering the conditions which obtain in small specimens.

Due to the fact that volume equilibrium is reached very slowly when hydrating cement is exposed to dry air, the hydration progresses simultaneously with the contraction. The gel structure grows and becomes more stable while the dimensional relations between the gel and the crystalline components are changing. If water soaking follows a drying cycle, the original volume of a neat cement specimen is not likely to be restored, because part of the gel structure has formed and hardened while the volume was less than the original. It would be expected that the original volume would be most nearly restored when the hydration has been the most nearly complete before drying began. Numerous test results show that water soaking of well-cured neat specimens causes them to expand very nearly to their original length, while poorly cured specimens retain much of their shrinkage.

In the case of concrete, the expansion upon resaturation may be sufficient to increase the volume beyond the original. The paste attempts to shrink upon drying, but the aggregate particles attempt to remain constant in volume. The shrinking paste attempts to compress the aggregate

TABLE I.—SHRINKAGE OF NEAT CEMENT BARS.

Water-Cement Ratio	Change in Length, millionths per unit			
	35 days	2 months	3 months	4 months
0.30	786	1514	1822	1998
0.40	1037	2205	2530	2847

NOTE.—Specimens $1\frac{1}{2}$ by $1\frac{1}{2}$ by 12-in. bars, moist cured for 28 days, then in air of 50 per cent relative humidity at 80 F.

TABLE II.—SHRINKAGE OF NEAT CEMENT, MORTAR, AND CONCRETE.

Mix by Weight	Aggregate Size	Voids,[a] per cent	Change in Length,[b] millionths per unit			
			35 days	2 months	4 months	Oven Dry[c]
WATER-CEMENT RATIO 0.30						
Neat	(none)	9.5	786	1514	1998	3400
1:2.85	No. 48 to No. 14 sieve	38.8	730	970	1091	1740
1:2.85	$\frac{3}{8}$ to $\frac{3}{4}$ in.	5.9	345	400	414	535
WATER-CEMENT RATIO 0.40						
Neat	(none)	13.2	1076	2107	2847	4647
1:2.85	No. 48 to No. 14 sieve	26.9	743	1016	1129	1492
1:2.85	$\frac{3}{8}$ to $\frac{3}{4}$ in.	6.0	353	431	443	458

[a] Voids computed as percentage absorption by volume after drying 4 months at 50 per cent relative humidity.

[b] Change in length measured on $1\frac{1}{2}$ by $1\frac{1}{2}$ by 12-in. bars, moist cured for 28 days, then at 50 per cent relative humidity.

[c] Oven-dry values measured on bars oven dried after 4-month readings taken.

TABLE III.—SHRINKAGE OF NEAT CEMENT, MORTAR, AND CONCRETE EVACUATED SERIES.[a]

Mix by Weight	Aggregate Size	Voids, per cent	Change in Length, millionths per unit			
			35 days	2 months	Oven Dry	Saturated[b]
WATER-CEMENT RATIO 0.30						
Neat	(none)	9.6	−705	−1420	−3400	−708
1:2.85	No. 48 to No. 14 sieve	41.0	−759	−988	−1492	−622
1:2.85	$\frac{3}{8}$ to $\frac{3}{4}$ in.	6.2	−325	−404	−458	+182
1:2	No. 48 to No. 14 sieve	34.9	−809	−1009	−1527	−722
1:2	$\frac{3}{8}$ to $\frac{3}{4}$ in.	5.7	−293	−404	−459	+123
WATER-CEMENT RATIO 0.40						
Neat	(none)	13.8	−1037	−2205	−4195	−1212
1:2.85	No. 48 to No. 14 sieve	28.7	−802	−1115	−1805	−727
1:2.85	$\frac{3}{8}$ to $\frac{3}{4}$ in.	7.3	−374	−460	−555	+72
1:2	No. 48 to No. 14 sieve	11.3	−548	−903	−1229	−537
1:2	$\frac{3}{8}$ to $\frac{3}{4}$ in.	7.0	−317	−388	−450	+158

[a] Mixtures placed under vacuum for 2 min. before casting in forms.

[b] Saturated values measured on bars boiled in water after oven drying, age 2 months.

particles, while the aggregate particles attempt to prevent the paste from shrinking. Thus, the bulk of the paste is in a state of tension and the aggregate particles are in a state of compression. Since the tensile strength of the paste is limited, it cracks between large particles of aggregate. But before the internal cracking occurs, the paste has been subjected to large forces which may have deformed it plastically. Upon resaturation, the paste may then expand into a slightly modified shape which will not perfectly fit the spaces between the aggregate particles. Furthermore, after internal cracking occurs, the cracks may tend to heal with gel or calcium hydroxide. When such effects predominate, there may be an expansion beyond the original volume. The test results in Table III illustrate this expansion.

A further effect of the dilution of paste with aggregate will be the entraining of air and the production of additional voids not present in the neat paste. The voids not only permit the more ready escape of moisture from the mortar or concrete, but they may also permit the paste to shrink more freely. Mortars and concretes are, therefore, expected to shrink more than would be computed from relative volumes and dimensions of paste and aggregate.

A study of such data as are available indicates a greatly increased shrinkage of concrete over what would be expected from the dimensions of the paste and aggregate. In a mix of one part of cement to about three parts by weight of gravel aggregate, approximately uniform in size, the volume of the paste was only about that necessary to fill the voids in the aggregate. In this case, the aggregate particles were almost in contact with one another. Although a random section cut through the specimen did not cut every particle where it was closest to another particle, a line could be found that ran almost straight from end to end along which every particle was only slightly separated by paste from its neighbor. Along such a line, the paste separating the aggregate made up less than 5 per cent of the length of the specimen. Assuming that the paste between the particles along this line shrinks exactly as it did in the neat paste specimens, and assuming that the aggregate particles remain constant in volume, the mortar or concrete would contract less than 5 per cent as much as the neat paste. Test results show that the shrinkage was a great deal more than this.

In Table II are shown values for the contraction of the plain specimens of neat paste and corresponding specimens of similar paste diluted with gravel aggregates of two different sizes. These specimens are unusual in composition, but serve well for illustration. Comparing the shrinkages at all ages up to four months, it may be seen that for both sizes of aggregate and for all ages, the shrinkage of the concrete or mortar is far in excess of 5 per cent that of the neat paste. It will be seen, however, that the general tendency is for the neat paste to continue shrinking at a more rapid rate than the mortars or concretes.

Table II shows further that the shrinkage varies greatly with the size of the aggregate; the larger the aggregate, the less the shrinkage. Disregarding for the moment the reasoning presented above, the shrinkage for small aggregate and early ages is even greater than would be expected were the paste assumed to be a liquid having a volume change equal to the amount observed on neat bars. The extremely high percentage of voids in the mortar in this case has magnified their effect and these specimens are useful, therefore, in investigating the manner in which voids increase the shrinkage.

In another series, an attempt was made to control void contents in the various specimens by placing the freshly mixed materials under vacuum before casting in forms. The results of tests on specimens made in this manner are given in Table III, previously mentioned. The mere application of a vacuum proved to have little effect on the void content and therefore the results serve mainly to substantiate those in Table II. Further tests are being made on specimens placed under vacuum and vibrated immediately after casting.

In order to determine whether the more rapid escape of moisture or some other effect was responsible for the apparent relation between voids and shrinkage, the amount of moisture remaining in specimens which had been drying at 50 per cent relative humidity up to the age of 2 months was determined. The specimens were first oven-dried at 110 C. (230 F.) to determine the amount of water still remaining that could be driven off at this temperature. Length measurements were also made to determine whether or not the ultimate shrinkage obtained by oven-drying would bear any simple relation to the geometry of the aggregate and paste. It was found in the oven-drying that the neat cement shrunk many times more than did the mortar or concrete, but that the total mortar and concrete shrinkages were nevertheless greater than could be expected from considerations of dimensions of aggregate particles and paste alone (see Tables II and III).

For the mix of 1:2.85, the percentage of moisture (by weight of cement) driven off by oven drying was least for the mortars, intermediate for the concretes, and greatest for the corresponding neat cements. This is in line with the observation that the mortar shrinkage up to this time was much greater than expected and the concrete shrinkage somewhat greater than expected. For the mortars and concretes of 1:2 mix, however, the percentage of moisture driven off was not far different from that for corresponding neat cement. In some cases, the neat cement even showed the greatest loss of moisture. Since the mortar of 1:2 mix also showed a shrinkage abnormally high for the amount of cement paste it contained, it is concluded that the more rapid escape of moisture is not the principal means by which voids increase the shrinkage of small specimens.

The most probable manner in which voids increase shrinkage is in cushioning the aggregate particles. Petrographic examinations of thin sections of mortars and concretes described above showed greater porosity around the aggregate particles than in the paste. There did not appear to be a continuous opening around the particles, but rather close contact of the paste with the high points on the irregular aggregate surfaces. If the aggregate particles are covered with air pockets, the likelihood is introduced of large plastic or elastic deformations in the thinner portions of the cement paste.

Plastic deformation of the paste films separating the aggregate particles may be the most important factor in concrete shrinkage. Plastic flow studies indicate that the major cause of the continued yielding of concrete under load is the expulsion of moisture from the hydrated cement. If conditions are such as to facilitate ready expulsion of moisture, plastic flow is increased. Although the bulk of the cement paste in concrete that is contracting is under tension due to the restraining effect of the aggregate, that in the thin films separating the aggregate particles is compressed with the aggregate. It is the paste in these thin films that largely controls the shrinkage. Conditions are usually good for the thin films to yield plastically, because the moisture that is expelled may go either into the voids in the aggregate or into the voids on the surface of the aggregate. Furthermore, stresses in the paste films may be concentrated at the points of contact with the aggregate particles. Such a view does not conflict with the observed tendency for the mortars and concretes to shrink relatively little at the later ages, when the paste films have become more rigid and internal cracks have begun to develop in the body of the paste.

The compression of aggregate particles in concrete probably has a lesser influence on shrinkage except when the aggregates are greatly lacking in rigidity. A certain amount of compression of aggregate particles seems inevitable. It would be expected that the smaller particles could be more easily compressed than the larger particles, thus accounting for the greater shrinkage in the case of the smaller aggregate. But the shrinkages are so great, in view of the dimensions of paste and aggregate particles, as to make it highly improbable that there would be sufficient tension in the paste to compress the particles the necessary amount. Furthermore, microscopic examinations reveal minute cracks between the larger particles of aggregate which rule out the possibility of large tensions in the paste.

If plastic deformation of the paste films separating closely adjacent particles of aggregates is a major factor in concrete shrinkage, it should explain the difference in shrinkage of concretes containing aggregates of different types. For example, tests have shown high shrinkage for a local siliceous gravel, but low shrinkage for a mixture of limestone and quartz gravel and for crushed limestone and crushed dolomite. The shape of particles seems to be of minor importance, because high shrinkage has been obtained when large pieces of the local siliceous gravel were crushed and used to replace identical sizes of the gravel. The rigidity of the aggregate appears to be of minor importance also, because lowest shrinkage was obtained for an aggregate with a fairly low modulus of elasticity. If the paste-deformation hypothesis above is tenable, the local siliceous aggregate must either be more porous than the others or it must entrain more air voids on the surfaces of the particles.

Absorption tests were made on the four aggregates mentioned and it was found that there was a close correlation between the absorption of aggregate and the shrinkage of concrete. Dolomite showed the lowest absorption (about 0.4 per cent) and this material gave low concrete shrinkage even though the particle shape necessitated higher water content. The local siliceous gravel which gave the highest shrinkage had the highest absorption (about 1 per cent).

Microscopic examinations of thin sections of concretes containing crushed dolomite and local siliceous gravel indicated poorer contact between aggregate and paste for the siliceous gravel. While this evidence was not entirely conclusive, since it depended upon the judgment of the observer, it helps to substantiate the hypothesis suggested. There remain to be determined the particular properties of aggregates which are conducive to tight bond with cement paste.

SHRINKAGE OF CONCRETE IN LARGER MASSES

It has been shown that the escape of moisture which is in loose chemical combination with the hydrated cement causes shrinkage, but that this moisture escapes very slowly. In order to determine the shrinkages which will obtain in concrete structures, tests on small specimens are not sufficient. On the other hand, measurements on large specimens exposed to drying on all surfaces are complicated by stresses and cracking, because the shrinkage is more rapid near the surface than in the interior. To obviate these difficulties, specimens were made and allowed to dry from one surface only, thus reducing stresses and preventing cracking. Shrinkages of various parts of these specimens indicated to what extent concrete in structures might contract if there were no stresses or surface cracking.

These specimens were prisms of 6 in. square cross-section, with lengths of 6 in. and 22 in. They were cast in very thin, soft copper jackets to seal all surfaces except one of the ends of each. They were kept in a moist room for 28 days and then placed in a drying room maintained at 50 per cent relative humidity and 80 F. As they dried from the exposed surface, all moisture was required to travel in one direction toward that surface to escape. Shrinkages were measured with a 5-in. strain gage on two opposite surfaces, in two directions at right angles, and at different distances from the exposed surface. There were 26 gage lines on the longer prisms and 8 on the shorter prisms. Four of the longer and seven of the shorter prisms were made, involving variations in cement and aggregate. The results obtained on only one of each are presented here, since they are fairly typical.

The observed shrinkages for comparable prisms of the two lengths are given in Table IV. It may be observed that the shrinkages measured on the longer prism are so small as to be negligible on all lines of the prism except that which is near the exposed surface and parallel to it. The gage lines nearest to the exposed surface but perpendicular to it show no shrinkage but rather a slight expansion up to the age of 3 months. While it was first concluded that this expansion indicated a tendency of concrete to shrink differently in different directions as related to the direction of moisture travel, further studies indicated this to be unlikely. The most probable significance of the expansion is that there must be an abrupt change in shrinkage from the exposed face inward. This results in shearing stresses which tend to curl the end of the specimen and make it concave,

TABLE IV.—INFLUENCE OF SIZE OF SPECIMEN, DISTANCE FROM EXPOSED SURFACE, AND DIRECTION OF MOISTURE TRAVEL UPON SHRINKAGE OF CONCRETE.

Age	Change in Length, millionths per unit									Change in Length of Small Bars for Comparison (1½ by 1½ by 12 in.)
	Parallel to Moisture Movement				Perpendicular to Moisture Movement					
	3.5 in. from Drying Face	8.5 in. from Drying Face	13.5 in. from Drying Face	18.5 in. from Drying Face	1 in. from Drying Face	6 in. from Drying Face	11 in. from Drying Face	16 in. from Drying Face	21 in. from Drying Face	
6 BY 6 BY 22-IN. PRISM										
28 days	−26	−4	−2	−6	+46	+20	−22	−4	+2	+30
35 days	+28	−10	−18	−28	−106	+2	−24	−36	−26	−323
2 months	+50	0	−34	−24	−156	+2	−32	−8	+2	−454
3 months	+22	+18	0	−10	−276	+8	−20	−14	−10	−499
4 months	+8	0	−10	−20	−306	−8	−12	−24	−18	−516
6-IN. CUBE										
28 days	+14	....	....	....	+14	+10	....	....	....	+30
35 days	+32	....	....	....	−52	0	....	....	....	−323
2 months	−28	....	....	....	−192	−64	....	....	....	−454
3 months	−26	....	....	....	−266	−34	....	....	....	−499
4 months	−56	....	....	....	−286	−56	....	....	....	−516

NOTE.—All specimens made of concrete containing siliceous gravel aggregate ¾-in. maximum size; 5.25 sacks commercial cement per cubic yard; 0.70 water-cement ratio by weight; prism and cube covered by thin copper jackets on all faces except one; 6-in. square surface exposed to drying in either case; small bars exposed on all surfaces; specimens moist cured for 28 days, then in air of 50 per cent relative humidity at 80 F.

expanding the edges that are perpendicular to the exposed surface, thus influencing the measurements.

In the case of the shorter prism, there is some shrinkage in all parts of the specimen, even in the direction of moisture travel. The shrinkages near the surface of both the smaller and the larger prism were about equal, but the larger prism showed no shrinkage elsewhere. It is suggested that the greater total amount of water in the larger prism is responsible for the difference and that in large masses moisture is fed toward the surface from the underlying concrete so as to prevent or delay substantial contraction except near the exposed surface.

The outstanding fact revealed by the data in Table IV is that for short periods the shrinkage of small bars is far greater than that of larger specimens. Up to the age of four months, the average shrinkage of a prism which simulates a slab 6 in. thick drying from one surface, or 12 in. thick drying from two surfaces, is shown to be only about one-third that of a small bar $1\frac{1}{2}$ in. in cross-section. For the same age, the average shrinkage of a prism which simulates a slab almost 2 ft. thick drying from only one surface is shown to be almost negligible, although the shrinkage 1 in. from the surface is more than half as great as the shrinkage of small bars. Under ordinary climatic conditions, the average shrinkage of structural members 1 ft. or more in thickness probably never would approach that of the small bars. Furthermore, in very massive structures, drying shrinkage should never be important except for that concrete within a few inches of the surface.

SUMMARY

In the hope of explaining the wide differences in concrete shrinkage obtained in various aggregates and conditions, a number of unusual test results have been presented and discussed. Certain hypotheses have been suggested, but the test results are not yet complete enough to justify the drawing of final conclusions. The aim has been to provide a tentative picture to serve until mechanics of shrinkage are more fully understood.

To summarize the results and discussions which have been presented, the factors which are believed to affect concrete shrinkage are divided into a major and a minor group. The data on some of the factors are meager as yet and some factors of importance have undoubtedly been neglected. Tests now in progress should help to increase the accuracy of the classification.

The factors believed to have a major influence on concrete shrinkage are tentatively listed as follows:

1. Amount and quality of gel in the hydrated cement; this factor is governed by water content, cement fineness and composition, and time of moist curing.

2. Amount of cement paste in the concrete mix.
3. Porosity of concrete.
4. Surface character of the aggregate particles as regards the ability to entrain air.
5. Porosity of aggregate.
6. Distance from an exposed surface (this factor probably varies with porosity).
7. Size of concrete specimen (this factor also probably varies with porosity).
8. Plastic flow of cement paste (controlled somewhat by strength of paste).
9. Humidity to which concrete is exposed.
10. Size of aggregate, mainly as it affects water content, voids, and number of surfaces of contact between paste and aggregate.
11. Time of exposure to drying (determined by duration of dry season for service structures).

The factors believed to have a minor influence on concrete shrinkage are tentatively listed as follows:

1. Rigidity of aggregate (within limits ordinarily encountered).
2. Shape of aggregate particles.
3. Mineralogical composition of aggregate (siliceous as compared with calcareous aggregates).
4. Percentage of largest-size particles in aggregate.
5. Gradation of aggregate, as it affects porosity and closeness of particles.

Acknowledgment:

The author takes pleasure in acknowledging his indebtedness to Jerome Raphael for making most of the measurements reported in Tables I, II and III; to Dr. Levi Brown for making and examining thin sections of the specimens; and to F. R. McMillan and Dr. R. H. Bogue of the Portland Cement Association for providing pertinent data and contributing most helpful thoughts.

DISCUSSION

Mr. W. C. Hanna.[1]—I should like to ask Mr. Carlson whether the information developed would account for some of these large, deep pattern cracks such as are sometimes found on the surface of concrete work where there is a fairly thick mass or wall? Take, for example, a concrete wall built with siliceous aggregate, three or four feet thick and made with the usual amount of water used in placing concrete, where the forms are stripped at a fairly early date, not cured very much with water, and then exposed to a dry cold wind.

Mr. R. W. Carlson.[2]—If the surface cracks in concrete are due to drying alone, the cracks are nearly always very close together, usually of the order of two or three inches, because the drying extends such a short depth into the concrete that when one crack forms, it relieves the stress for only a short distance away from that crack, so another one must form quite close. In the case of the larger masses, when the forms are removed, the surface cools while the center concrete may still be warming; consequently there is a development of tension which may extend a few feet into the concrete. If you have superimposed the tension right at the surface due to drying shrinkage, you exceed the tensile strength of the concrete at the surface and start a crack which might not otherwise form. Once the crack is formed, it goes to a considerable depth, relieving the small but extensive tension due to the temperature changes. That would probably account for the pattern cracks that are of sufficient size to be seen by casual observation.

The Chairman (*Mr. J. C. Pearson*[3]).—I should like to ask Mr. Carlson a question. I believe you stated that there was a close correlation between the absorption of the aggregate and the shrinkage of concrete. Would you expect this relation to hold generally—that is, would any aggregate low in absorption tend to make concrete of low shrinkage?

Mr. Carlson.—At present we think that either the absorption of the aggregate or the character of the surface of the aggregate as regards its ability to produce voids on its surface must be the cause of the greater shrinkage. In any concrete the aggregate does not prevent the paste from shrinking as well as would be expected. An aggregate which has a low absorption, and probably one which has a surface condition which makes the paste fit closely to the aggregate, would be the one which would give the low shrinkage.

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[1] Chief Chemist, California Portland Cement Co., Colton, Calif.
[2] Assistant Professor of Civil Engineering, Massachusetts Institute of Technology, Cambridge, Mass.
[3] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

MR. BEN MOREELL.[1]—There are several statements made by Mr. Carlson which are similar to statements previously made by Freyssinet to explain volume changes in concrete. One of the statements made by Mr. Carlson I consider to be quite significant, namely, that the shrinkage increased with the porosity of the aggregate. That would seem to indicate that there was a penetration of the water into the aggregate and a resulting action of capillary forces to reduce the volume of the aggregate particles. Freyssinet showed that the capillary attractions are of the order of 30,000 to 40,000 lb. per sq. in. If the aggregate particles are subjected to forces of that magnitude by virtue of the capillary attraction, that is, the attraction of the water in the capillary canals, there must of necessity be a considerable reduction in the volume of the aggregate. I cannot conceive of an aggregate being subjected to forces of that magnitude without yielding, because, after all, the aggregates are plastic materials; that is, we can conceive of them as being partly elastic and partly plastic. That would explain the fact that the concrete made with aggregates that are more porous would show the greatest shrinkage. The writer had occasion to make a translation of M. Freyssinet's theory several years ago and has a few extra copies available for distribution.

MR. P. H. BATES[2] (*by letter*).—While many studies have been made in recent years on the shrinkage of concrete, especially in the United States, the fundamental nature of the concrete, and particularly of the hydrated cement, has been left distinctly in the background. It is hoped that this paper by Mr. Carlson will serve to arouse the interest of many concerned with concrete shrinkage in the fundamentals of the subject, and will further serve to bring investigators into a field which has been emphasized many times, but which in the United States has been lost sight of for some time.

In France, for a period of about seven years, this subject has been to the fore through the efforts of Freyssinet, the designer of the famous bridge at Plougastel, which stands to date as the concrete bridge containing the longest span arches in concrete anywhere. One of his most recent publications on this subject appeared in *Science et Industrie* for January, 1933. This was translated from the original transcript by Commander Ben Moreell, Civil Engineer Corps, U. S. Navy, Washington, D. C. For those who would be interested in Freyssinet's presentations of this subject, which to date unquestionably are the most fundamental that we have, may I quote from this translation by Commander Moreell because it shows very briefly how Freyssinet approached the subject and gives at the same time some of his publications:

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[1] Commander, Civil Engineer Corps, U. S. Navy, Bureau of Yards and Docks, Navy Dept., Washington, D. C.

[2] Chief, Clay and Silicate Products Division, National Bureau of Standards, Washington, D. C.

"Having available the first results of the experiments undertaken in connection with the construction of the bridge at Plougastel, there occurred to me intuitively, the fundamental law governing the deformation of cements, namely, that this deformation is the result of an equilibrium between the exterior forces and the internal forces, the latter being variable with the conditions of the surrounding medium. The first observations were reported in 1926, in a communication to the 'Commission Technique de la Chambre Syndicale des Constructours on Beton Arme'; and later, in 1928, at the Congré of Vienna. I later extended the experiments, the results of which I was able to correlate with the general principles of thermo-dynamics.[a]"

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[a] This theory was presented at the School of Engineers at Lausanne in October, 1929, later at a meeting of the Association Internationale des Materiaux de Construction. A résumé of the experiments was inserted in the reports of the Congress of Liege on Reinforced Concrete of 1930; the discussions at the Congress of Liege 1930 and Congress of Paris 1932 resulted in several publications of parts of the theory, of which the latest is that in the *Annales des Ponts de Chaussées* (III–1932). (Note by the translator.—A translation of the article which appeared in the *Annales des Ponts et Chaussées* is available.)

SHRINKAGE OF CONCRETE

By Inge Lyse[1]

Synopsis

This paper presents the results of an investigation of the use of ordinary 3 by 6-in. concrete cylinders for volume change observations and of observing these changes by a simple apparatus consisting of an Ames dial attached to a metal base, and of a study of some of the factors which contribute to volume change in concrete. The factors investigated were quality and quantity of the cement paste used, length of moist curing, and different cements and aggregates.

The 3 by 6-in. cylinders were found to serve well for shrinkage observations and the measuring apparatus used gave satisfactory results. The quality of the paste showed little effect upon the shrinkage, while the quantity of the paste contributed approximately in proportion to its percentage of the volume of the concrete. The length of moist curing had practically no effect upon the shrinkage. The high-early-strength portland cement produced more shrinkage than did the standard portland cement, particularly in lean mixes, but in terms of shrinkage per unit of strength, the high-early-strength cement proved superior. Fine aggregate showed more effect upon shrinkage than did coarse aggregate.

Introduction

The problem of volume changes in concrete caused by variation in its moisture content has been given much experimental study by numerous investigators. An excellent review of present knowledge of these volume changes was recently presented in a paper by Davis.[2] This paper contains such a complete bibliography of the more important publications in this field, that it is unnecessary to present but later references here. One of the most important recent publications is by the late Professor Slater,[3] in which is presented a summary of the results of the elaborate investigation carried out by the Portland Cement Association over a number of years.

The investigation undertaken at the Fritz Engineering Laboratory of Lehigh University had as its aims the study of a simplification in volume change experiments, and of some of the fundamental causes of shrinkage of concrete. For the purpose of simplifying the experimental work, ordinary 3 by 6-in. concrete cylinders were used as test specimens, both for shrinkage measurements and for compression tests. The particular shrinkage factors investigated were quality of cement paste, quantity of cement paste, length of moist curing, and a few different types of cement and aggregate. Certain of these factors have received but scant observations in the past, and since they were considered to be some of the most significant factors in the quality-giving elements of concrete, they were selected for this study.

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[1] Research Associate Professor of Engineering Materials, Lehigh University, Bethlehem, Pa.

[2] R. E. Davis, "A Summary of the Results of Investigations Having to do with Volumetric Changes in Cements, Mortars and Concretes, Due to Causes Other than Stress," *Proceedings*, Am. Concrete Inst., Vol. 26, p. 407 (1930).

[3] W. A. Slater, "Designing Concrete for High Strength, Low Permeability and Low Shrinkage," *Proceedings*, Internat. Assn. Testing Mats., Vol. 1, p. 1000 (1931).

OUTLINE OF TESTS

The test program was divided into five groups, each group devoted to one certain factor. Group 1 dealt with the effect of the quality of the cement paste of the concrete. This quality was varied from a cement-water ratio by weight of 1.0 which represents a very lean concrete mix, to a ratio of 2.5 which represents a very rich mix. Four cement-water ratios by weight were used (1.0, 1.5, 2.0, and 2.5) and two consistencies, one having slumps between 1 and 3 in., and one between 6 and 9 in. Thus the range included all concrete mixes used in ordinary construction. Two sets of specimens were made, one for compression test at the age of 7 days, that is, at the time the other set of specimens was removed from the moist room and placed in dry storage for observation of change in length. Each set of specimens consisted of three 3 by 6-in. cylinders, which were made, capped, cured, and tested for strength in accordance with standard practice. All specimens of each group were made on the same day (a deviation from the usual practice) so that they would receive identical treatment throughout the investigation. All mixes of relatively dry consistency had a water content of 38 gal. per cu. yd. of concrete, regardless of cement-water ratio, and all the mixes of wet consistency had a water content of 45 gal. per cu. yd. The information regarding the concrete in group 1 is given in Table I. The cement used was a standard portland, cement A of Table IV.

Group 2 was designed for the study of the effect of the quantity of paste. Two mixes were used, in one of which the cement-water ratio was 1.0, and in the other 2.0. The quantity of paste was so controlled that concrete varying from a very dry to a relatively wet consistency was secured. The percentages of paste for the lean mix (cement-water ratio of 1.0) were 24, 26, 28, and 30. The corresponding percentages for the rich mix were 30, 32, 34, and 36. The information on these mixes is given in Table II. As in group 1, two sets of specimens were made, one for compression test at the time of removing the specimens from the moist room, which was at the age of 7 days, and one set for shrinkage observation. The cement used was cement A of Table IV.

Group 3 was made for the study of the effect of the length of moist curing on the shrinkage of concrete. Two mixes had standard portland cement (cement A of Table IV) and two had high-early-strength portland cement (cement B of Table IV). The cement-water ratios of the two mixes

TABLE I.—CONCRETE USED IN GROUP 1.

Batch	Cement-Water Ratio by Weight	Water Content, gal. per cu. yd.	Concrete Mix by Weight	Paste, per cent	Slump, in.	7-day Compressive Strength, lb. per sq. in.
No. 1	1.0 (1.22)	38	1:4.16:6.24	24.8 (21.4)	$\frac{1}{4}$	470
No. 2	1.0 (1.22)	45	1:3.31:4.96	29.4 (25.4)	6	480
No. 3	1.5	38	1:2.65:3.98	27.8	$2\frac{1}{2}$	1510
No. 4	1.5	45	1:2.08:3.12	33.0	8	1240
No. 5	2.0	38	1:1.91:2.86	30.8	$3\frac{1}{4}$	3030
No. 6	2.0	45	1:1.48:2.22	36.6	9	2770
No. 7	2.5	38	1:1.47:2.20	34.0	2	4320
No. 8	2.5	45	1:1.12:1.68	40.2	7	4680

TABLE II.—CONCRETE USED IN GROUP 2.

Batch	Cement-Water Ratio by Weight	Paste, per cent	Water Content, gal. per cu. yd.	Concrete Mix by Weight	Slump, in.	7-day Compressive Strength, lb. per sq. in.
No. 9	1.0	24	36.7	1:4.35:6.53	$\frac{1}{4}$	590
No. 10	1.0	26	39.8	1:3.92:5.88	2	570
No. 11	1.0	28	42.8	1:3.52:5.28	4	490
No. 12	1.0	30	45.9	1:3.20:4.80	$6\frac{3}{4}$	490
No. 13	2.0	30	37.0	1:1.99:2.98	$1\frac{1}{4}$	3240
No. 14	2.0	32	39.4	1:1.82:2.73	$4\frac{3}{4}$	3050
No. 15	2.0	34	41.8	1:1.67:2.51	6	3410
No. 16	2.0	36	44.3	1:1.53:2.30	$8\frac{3}{4}$	3490

TABLE III.—CONCRETE USED IN GROUP 3.

Batch	Type of Portland Cement	Cement-Water Ratio by Weight	Concrete Mix by Weight	Slump, in.	Age at Removal to Dry Storage, days	Compressive Strength at Removal to Storage, lb. per sq. in.
No. 25	Standard	1.0	1:3.92:5.88	4	1	64
					3	226
					7	640
					28	1000
No. 26	Standard	2.0	1:1.82:2.73	$5\frac{1}{2}$	1	530
					3	1520
					7	2770
					28	5170
No. 27	High-early-strength	1.0	1:3.92:5.88	$4\frac{1}{2}$	1	178
					3	575
					7	940
					28	1500
No. 28	High-early-strength	2.0	1:1.82:2.73	$4\frac{1}{2}$	1	1480
					3	2930
					7	4600
					28	5600

TABLE IV.—CONCRETE USED IN GROUP 4.

Batch	Brand of Cement	Cement-Water Ratio by Weight	Paste, per cent	Concrete Mix by Weight	Slump, in.	7-day Compressive Strength, lb. per sq. in.	Fineness, per cent Passing No. 200 Sieve
No. 17	A	1.0	26	1:3.92:5.88	2	550	90.90
No. 18	B				4	810	97.30
No. 19	C				3¼	430	95.74
No. 20	D				4	860	99.86
No. 21	A	2.0	32	1:1.82:2.73	6½	3030	
No. 22	B				3½	4230	
No. 23	C				6½	2080	
No. 24	D				3¼	4100	

	Cement A	Cement B	Cement C	Cement D
SiO_2, per cent	19.96	19.52	22.44	20.92
Fe_2O_3, per cent	2.39	2.23	1.92	2.04
Al_2O_3, per cent	6.81	6.15	5.40	5.12
CaO, per cent	62.78	62.95	63.46	63.93
MgO, per cent	3.89	3.68	3.29	3.65
SO_3, per cent	1.84	2.48	1.66	2.29
Loss, per cent	1.83	2.53	1.35	1.52
Total, per cent	99.50	99.54	99.52	99.47

TABLE V.—CONCRETE USED IN GROUP 5.

Batch	Aggregate	Type of Aggregates		Cement-Water Ratio by Weight	Concrete Mix by Weight	Slump, in.	7-day Compressive Strength, lb. per sq. in.
		Fine	Coarse				
No. 29	No. 1	Bank 1	Gravel	1.0	1:3.20:4.80	7½	540
No. 30	No. 2	Bank 2	Gravel	1.0	1:3.20:4.80	6	510
No. 31	No. 3	Crushed Limestone 1	Gravel	1.0	1:3.20:4.80	4½	490
No. 32	No. 4	Crushed limestone 2[a]	Gravel	1.0	1:3.20:4.80	5	630
No. 33	No. 5	Bank 1	Crushed limestone	1.0	1:3.20:4.80	6	560
No. 34	No. 1	Bank 1	Gravel	2.0	1:1.52:2.28	8	2710
No. 35	No. 2	Bank 2	Gravel	2.0	1:1.52:2.28	7	2790
No. 36	No. 3	Crushed limestone 1	Gravel	2.0	1:1.52:2.28	6½	2810
No. 37	No. 4	Crushed limestone 2[a]	Gravel	2.0	1:1.52:2.28	4	2990
No. 38	No. 5	Bank 1	Crushed limestone	2.0	1:1.52:2.28	8½	3360

[a] Contained clay.

were 1.0 and 2.0. The ages at which shrinkage observations were first made and compression tests were made, were 1, 3, 7, and 28 days. As for all the other groups, the first day of curing consisted in leaving the concrete in the steel molds for this length of time. Subsequently curing was in a moist room of 100 per cent humidity and at 70 F. The information on the mixes used in group 3 is given in Table III.

Group 4 represents a study of the effect of the cement used in the concrete. Two brands of standard portland cements, cement A and C, and two brands of high-early-strength portland cements, cements B and D, were studied. Each brand of cement was used in two concrete mixes, one with a cement-water ratio of 1.0, the other with a cement-water ratio of 2.0. Moist curing was applied for 7 days. The information for this group is given in Table IV.

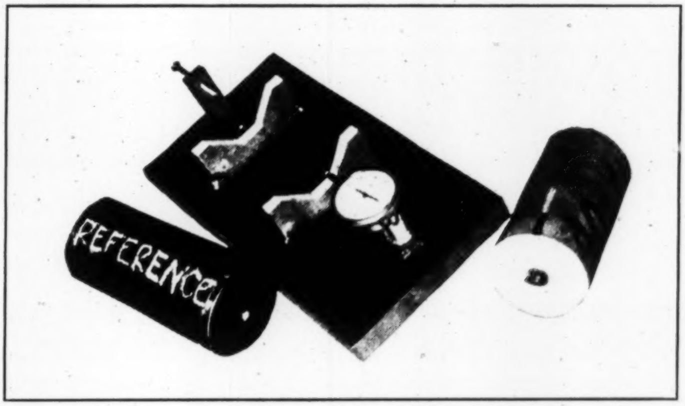

FIG. 1.—Measuring Apparatus and Weighing Balance.

Group 5 included five combinations of aggregates, containing four fine and two coarse aggregates. Two of the fine aggregates were natural bank sands, the other two were crushed limestone, one of which was relatively clean, while the other contained an appreciable amount of fine clay. The two coarse aggregates were natural siliceous gravel and crushed limestone. Mixes having cement-water ratios of 1.0 and 2.0 by weight were selected for the tests. The specimens were cured moist until tested at the age of 7 days. The information for this group is given in Table V.

The study presented in this paper was based on the shrinkage that took place during approximately $\frac{1}{2}$-yr. storage in dry air.

APPARATUS FOR SHRINKAGE OBSERVATION

The apparatus used for observing changes in length of the 3 by 6-in. concrete cylinders consisted simply of a steel base to which were welded two uprights; one of them held the screw which served as the fixed end and the other had a 0.0001-in. Ames dial attached for recording the length. Two V-shaped sections, welded to the base, served as guides for the cylinders. A black line, painted at one end of the cylinder, served to secure the same position during each observation. With this instrument the length of the cylinder could readily be determined to the nearest 0.0001 in. In order to secure the same method of observation the same operator took all readings. Immediately after the observation of length the specimen was weighed to the nearest gram for loss of water. A photograph of the measuring apparatus is presented in Fig. 1.

The 3 by 6-in. shrinkage specimens had stainless steel plugs in the center at each end. The specimen was so placed in the measuring apparatus that the plug at one end was resting against the screw which served as the fixed point, and the plunger of the Ames dial bore against the other plug.

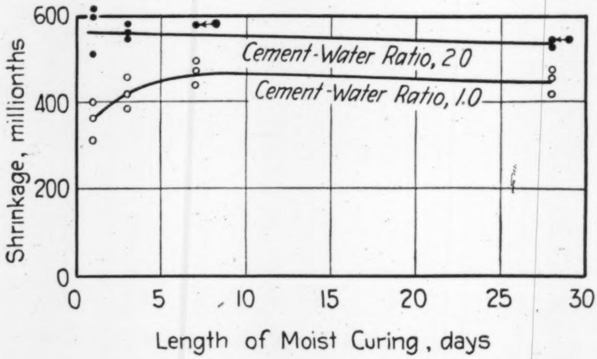

FIG. 2.—Variation Between Individual Specimens of Group 3.

These specimens proved very convenient for the shrinkage observation as well as for weighing. Since the plugs were set in ½-in. steel cubes cast in the concrete, the gage line was considered to be 5½ in. in working up the data.

An indication of the suitability of this apparatus and the 3 by 6-in. cylinders for volume-change observations is obtained from the study of variation in results between the individual specimens in a set. Figure 2 shows the individual results for the standard portland-cement specimens in group 3, which was a study of the effect of length of moist curing. It is noted that the individual variation is relatively small, particularly so when it is remembered that it is practically impossible to insure the identical amount of each constituent of the concrete in each of the companion specimens. The variation shown is therefore probably due more to the variation in the constituents of the concrete than to the method of observation. The results indicated that the very simple apparatus and the small specimens used in this investigation served well for observations of volume changes in concrete.

DRY STORAGE

The concrete cylinders which were observed for shrinkage were stored in a room with the temperature controlled fairly well to 80 F. No attempt was made to control the humidity, but a humidigraph gave the record of the relative humidity throughout the storage period. In general, the humidity varied between 40 and 60 per cent. Since all specimens of each group were made on the same day and for all groups, except group 3 on curing, were removed from the moist room at the same time, the variation

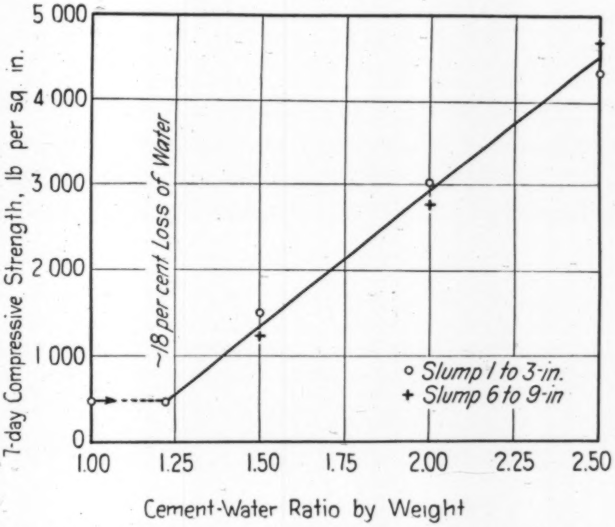

FIG. 3.—Relation Between Strength and Cement-Water Ratio of the Concrete in Group 1.

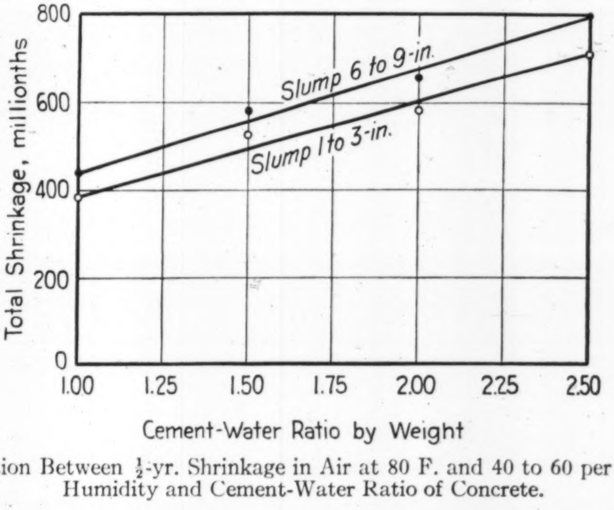

FIG. 4.—Relation Between $\frac{1}{2}$-yr. Shrinkage in Air at 80 F. and 40 to 60 per cent Relative Humidity and Cement-Water Ratio of Concrete.

in humidity did not have any effect upon the relative shrinkage results. The specimens in group 3 were not subjected to identical humidity conditions because of the difference in length of moist curing, but the results indicated that this possible variation in humidity probably did not affect the shrinkage appreciably.

EFFECT OF CEMENT-WATER RATIO—GROUP 1

The results of the specimens included in group 1 are presented in Figs. 3, 4 and 5. Figure 3 shows the relationship between the strength of the concrete at the age of 7 days, that is, at the time the volume change observations were started. It is noted that the relation between strength and cement-water ratio followed a straight line except for the very lean mix which had a cement-water ratio of 1.0. In this lean mix a part of the mixing water segregated from the concrete so that the actual water content became somewhat less than the total mixing water used in the batch. The strength relation in Fig. 3 indicates that the amount of segregated water was about 18 per cent of the total mixing water used.

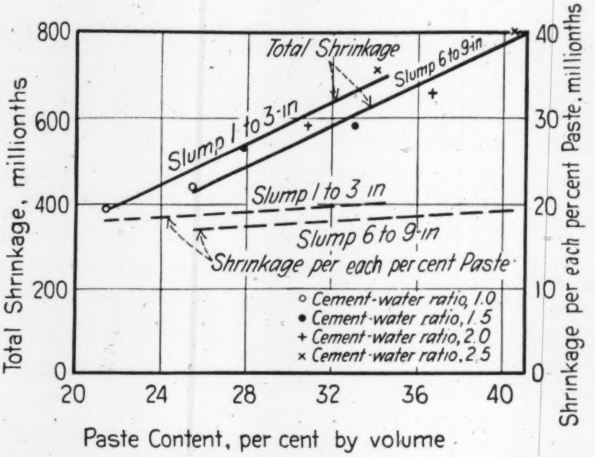

FIG. 5.—Effect of Paste Content on Shrinkage at 80 F. and 40 to 60 per cent Relative Humidity of Concrete in Group 1.

Figure 4 shows the relationship between shrinkage and cement-water ratio of the concrete. The shrinkage increased regularly with the increase in the cement-water ratio. The relatively dry mix of slumps from 1 to 3 in. showed less shrinkage than did the relatively wet mix of slumps from 6 to 9 in. Thus the paste content as well as the cement-water ratio seemed to affect the shrinkage.

In Fig. 5 the shrinkage of these mixes has been plotted against the percentage of cement paste in the fresh concrete. The percentage of paste for the mix having a cement-water ratio equal to only 1.0 has been corrected for the indicated loss in mixing water. No correction was applied to any of the other mixes. The shrinkage increased regularly with the increase in the paste content of the concrete. Thus the question arises: Is the shrinkage caused primarily by the quality of the paste as given by the cement-water ratio, or by the quantity of paste as given by the percentage of paste used? In order to obtain some information on this question, the shrinkage was studied in terms of unit of paste. The broken lines in Fig. 5 give the shrinkage values obtained when the shrinkage as given by the solid lines is divided by the amount of paste. The shrinkage per 1 per cent of paste is thus found to vary from approximately 16½ millionths for a cement-water ratio of 1.0 to 18 millionths for a cement-water ratio of 2.5.

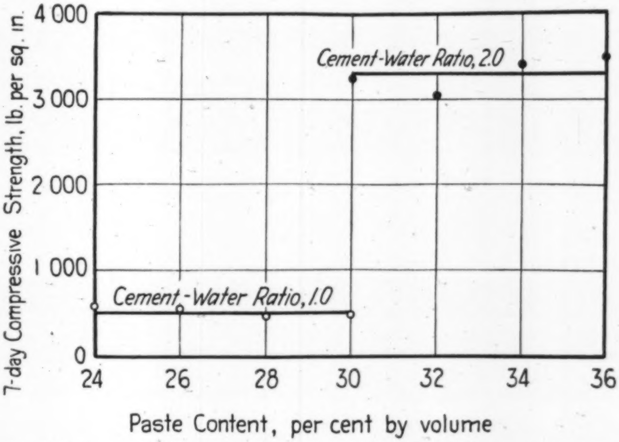

FIG. 6.—Effect of Paste Content on Strength of Concrete in Group 2.

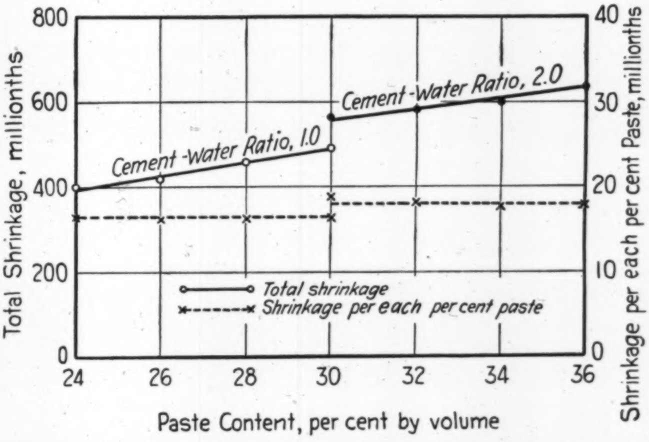

FIG. 7.—Effect of Paste Content on Shrinkage at 80 F. and 40 to 60 per cent Relative Humidity of Concrete in Group 2.

Although the cement content was increased 2½ times, the shrinkage per unit of paste increased only about 10 per cent. This indicates that for the standard portland cement used in this group, the shrinkage of the concrete was caused primarily by the amount of paste used in the mix. The quality of the paste had very little effect upon the shrinkage of the concrete. The slightly lower shrinkage for concrete containing paste of low cement content as compared to that for the higher cement content may also partially be due to the segregation of mixing water which took place more readily in the lean than in the richer mixes.

The information obtained in group 1 may therefore be summarized as follows: The shrinkage of the concrete was nearly proportional to the amount of paste used, the quality of the paste having little, if any, effect upon the shrinkage per unit of paste. The increase in shrinkage with increased richness of the mix was principally caused by the increase in the paste content.

EFFECT OF QUANTITY OF PASTE—GROUP 2

In group 2 two mixes were selected (cement-water ratios of 1.0 and 2.0) using standard portland cement. The quantity of paste was varied from 24 to 30 per cent for the lean mix and from 30 to 36 per cent for the rich mix. The results of the compression tests on companion cylinders at the age of 7 days are given in Fig. 6. The compressive strength remained practically constant, regardless of the quantity of the paste, as long as the quality of the paste remained constant. This is in agreement with the cement-water ratio strength law which states that the strength varies directly with the concentration of cement in the paste regardless of amount of paste.

Figure 7 shows the shrinkage of the concrete plotted against the percentage of paste used. The shrinkage increased quite uniformly with the increase in paste content. However, the rich mix with a cement-water ratio of 2.0 showed more shrinkage for 30 per cent nominal paste content than did the very lean mix with a cement-water ratio of 1.0. When the shrinkage is given per unit of paste, or per 1 per cent paste, each of the mixes showed a constant shrinkage. The lean mix showed a shrinkage of about 16½ millionths per 1 per cent paste, while the rich mix showed about 18 millionths. These values correspond very well with those obtained in Group 1 and indicate that within the range of ordinary mixes, that is, from cement-water ratios of 1.25 to 2.25, the shrinkage of concrete per unit of paste is practically constant, regardless of the quality of the paste. In order that the shrinkage for 1 per cent of paste be equal for the two mixes used, about 15 per cent of the mixing water in the lean mix would have to segregate. The results in group 1 showed that a segregation of this amount of mixing water might well have taken place. Thus both these groups indicate that the shrinkage of the concrete was rather independent of the cement-water ratio of the paste, or in other words, the shrinkage was independent of the cement content in the paste. The shrinkage of the concrete may thus be expressed approximately by the formula:

$$s = k \cdot p$$

where s is shrinkage, k is a constant indicating shrinkage per 1 per cent of

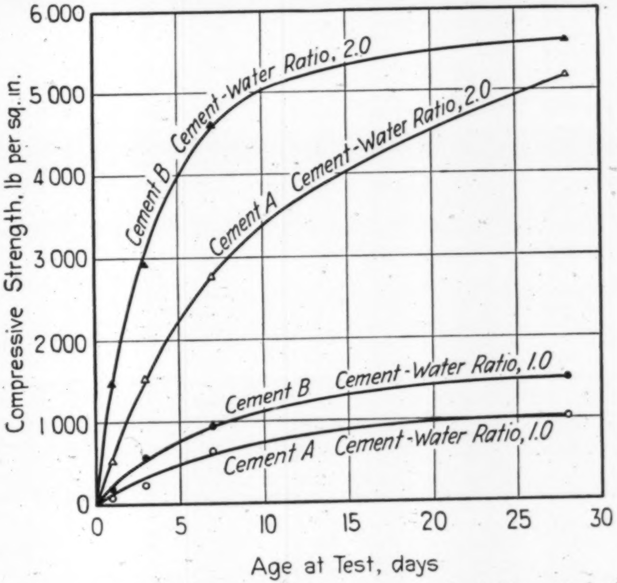

FIG. 8.—Effect of Length of Moist Curing on Strength of Concrete in Group 3.

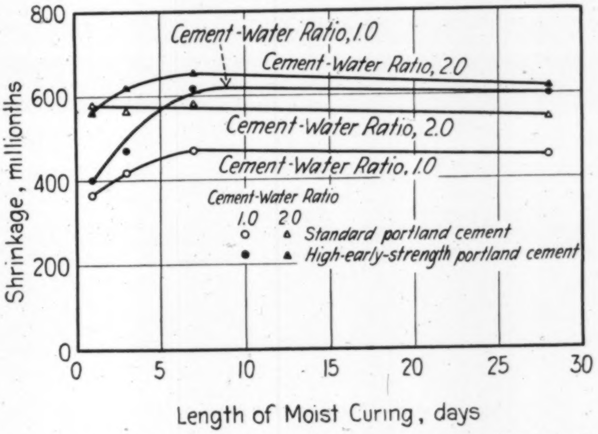

FIG. 9.—Effect of Length of Moist Curing on Shrinkage at 80 F. and 40 to 60 per cent Relative Humidity of Concrete in Group 3.

paste which depends upon the cement and aggregate used and upon the conditions of the tests, and *p* is percentage of cement paste in the concrete.

EFFECT OF LENGTH OF MOIST CURING—GROUP 3

The results of the strength tests are given in Fig. 8, and of the shrinkage tests in Fig. 9. It is noted that both the standard cement A and the high-early-strength portland cement B showed consistent increase in strength with increase in length of moist curing up to 28 days which was the maximum used in this investigation. The high-early-strength cement gave concrete strength considerably above that for the standard portland cement for both cement-water ratios used. For the lean mix the high-early-strength

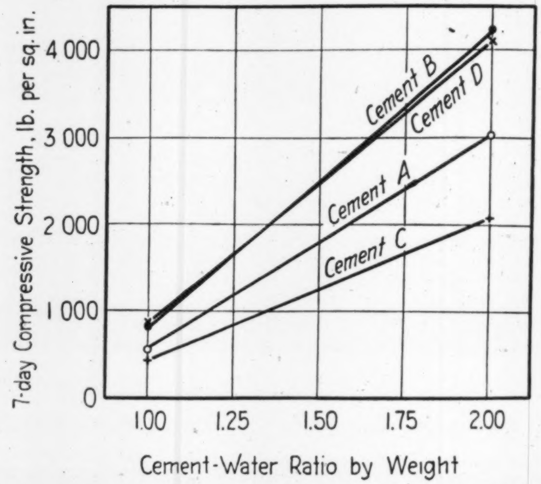

FIG. 10.—Effect of Cement on Strength of Concrete in Group 4.

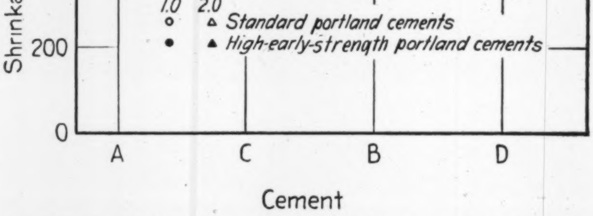

FIG. 11.—Effect of Cement on Shrinkage at 80 F. and 40 to 60 per cent Relative Humidity of Concrete in Group 4.

cement gave approximately 50 per cent greater strength than did the standard cement at all ages of test. For the rich mix the high-early-strength cement gave concrete strength about 200 per cent greater than standard portland cement at the age of 1 day, 100 per cent greater at 3 days, 70 per cent greater at 7 days and less than 10 per cent greater at 28 days.

The shrinkage of the concrete as presented in Fig. 9 indicated that the length of moist curing did not have any marked effect. The results showed a tendency of an increase in shrinkage with increased length of moist curing up to 7 days and then practically no change in the shrinkage with the increase in moist curing up to 28 days. The high-early-strength cement gave considerably greater shrinkage for the lean concrete than did the standard portland cement. However, the effect of the type of cement on the shrinkage of the rich concrete mix was relatively small.

EFFECT OF BRAND AND TYPE OF CEMENT—GROUP 4

In this group four cements were used, two of which were standard portland cements (of which one was cement A of the previous tests) and the remaining two were high-early-strength portland cements (of which one was cement B of the previous tests). The compressive strength at the age of 7 days is given in Fig. 10. The strength qualities of the two standard portland cements differed considerably, while the two high-early-strength cements were nearly equal.

Figure 11 shows the shrinkage of the concrete containing these four cements for the two concrete mixes used. The two standard portland cements showed approximately the same shrinkage both for the lean and rich mix. So also did the two high-early-strength cements. However, for the lean mix the shrinkage of the concrete containing high-early-strength cements was considerably greater than that of the concrete containing the standard portland cements, while for the rich mix the difference was not marked.

Comparing the shrinkage with the strength, it is noted that the high-early-strength portland cements produced higher strength for a given shrinkage than did the standard portland cements. Furthermore, the results indicated that for the high-early-strength cements, the shrinkage per unit of paste decreased with the increase in the cement-water ratio of the concrete—that is, the lean mix showed greater shrinkage per each per cent of paste than did the rich mix.

EFFECT OF TYPE OF AGGREGATE—GROUP 5

Numerous investigators have pointed out that the aggregate in the concrete has an important effect upon the volume change. This action of the aggregate may be due to the volume change of the aggregate itself, to effect of the aggregate on the volume change of the cement paste, or to a combination of the two. Observations by Schumann[1] on volume changes on various building stones showed a considerable variation between different materials. Although certain stones such as special types of limestone showed a shrinkage of only 80 millionths, finely grained sandstone showed as much as 1780 millionths, when dried in air for two weeks. Different types of rock material may thus shrink more than ordinary concrete.

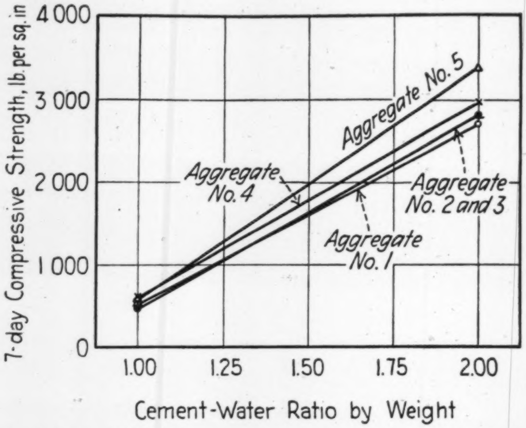

FIG.—12. Effect of Aggregate on Strength of Concrete in Group 5.

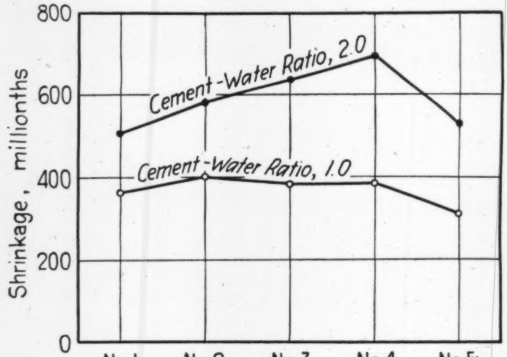

FIG. 13.—Effect of Aggregate on Shrinkage of Concrete in Group 5.

The results of the compressive strengths of the concrete containing the five different aggregate combinations in group 5, at 7 days are shown in Fig. 12. The compressive strength was affected very little by the different aggregates.

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[1] Schumann, "Report of Commission on Constancy of Volume of Portland Cement," Assn. German Portland Cement Mfrs. (1889), *Tonindustrie Zeitung*, Vol. 5, p. 184 (1881), and Vol. 13, p. 435 (1889). A summary of Schumann's results is given in the paper by R. E. Davis, "A Summary of the Results of Investigations Having to Do with Volumetric Changes in Cements, Mortars and Concretes, Due to Causes Other than Stress," *Proceedings*, Am. Concrete Inst., Vol. 26, p. 407 (1930).

The results of the shrinkage observations are presented in Fig. 13. The variation in shrinkage is relatively small for the lean mix with a cement-water ratio of 1.0. However, aggregates Nos. 1 and 5 which had the coarser bank sand showed less shrinkage than did the concrete containing the three other fine aggregates. For the rich mix with a cement-water ratio of 2.0, the effect of the aggregates was more marked. Again, aggregates Nos. 1 and 5 show about the same shrinkage, indicating that the two coarse materials used—siliceous gravel and limestone—did not produce appreciable difference in the volume change. Aggregates Nos. 2, 3, and 4 showed a considerably greater shrinkage.

For the aggregates included in this study, the results indicated that practically no difference occurred due to the two coarse materials used, while a considerable variation in shrinkage was caused by the different fine aggregates.

GENERAL DISCUSSION

The results of this investigation help to account for most of the apparent independent factors which have been found to affect the volume change of mortars and concrete. For standard portland cement the shrinkage of the concrete was found to be practically proportional to the paste content. Thus every factor which tends to decrease the paste content of the concrete will also tend to reduce the shrinkage. This accounts for the fact that, as previous investigators have found, lean mixes will shrink less than rich mixes, concrete will shrink less than mortars, and dry mixes less than wet mixes; also that concrete of low sand content will shrink less than concrete of high sand content and concrete with coarse sand will show less shrinkage than concrete with fine sand. Concrete having well-graded aggregate will shrink less than concrete having poorly graded aggregate and the addition of powdered admixtures would in general be expected to increase the shrinkage. Thus the paste content explains the reason for the effect of practically all factors which have been found to influence the volume change of concrete.

Since the shrinkage of concrete is due to the evaporation of its mixing water, the prevention of evaporation will prevent shrinkage. However, the amount of loss of mixing water did not give direct information of the amount of shrinkage of the concrete. For lack of space the data on loss in weight have been omitted from this paper.

SUMMARY

The most important shrinkage results of this investigation based on $\frac{1}{2}$ yr. dry storage of the concrete, may be summarized as follows:

1. The simple measuring apparatus used served well for volume change observations.

2. The 3 by 6-in. concrete cylinders proved easy to handle and gave dependable results both for compression tests and for shrinkage observations.

3. The quality of the cement paste, that is, the richness of the paste as given by the cement-water ratio, had practically no effect upon the shrinkage of concrete per 1 per cent of paste.

4. The shrinkage of concrete containing standard portland cement was practically proportional to the percentage of paste in the mix, regardless of the composition of the paste.

5. The length of moist curing up to 28 days had only small effect upon the shrinkage.

6. For lean mixes the high-early-strength portland cement produced considerably greater shrinkage than did standard portland cement, while for rich mixes the difference was inappreciable.

7. The shrinkage per unit of strength was less for high-early-strength than for standard portland cement.

8. For high-early-strength portland cement the shrinkage of the concrete per unit of paste was less for the rich mix than for the lean mix.

9. The fine aggregates showed greater effect upon the shrinkage than did the coarse aggregate.

10. For the standard portland cement used in this investigation (cement A), the shrinkage of the concrete may be expressed approximately by the formula:

$$s = k \cdot p$$

11. The results of this investigation are in agreement with previous investigations and the paste content theory explains many factors that have been found to contribute to the shrinkage of concrete.

DETERMINATION OF MODULUS OF ELASTICITY AND POISSON'S RATIO OF CONCRETE AT AGES OF FOURTEEN DAYS TO FOUR YEARS

BY L. H. KOENITZER[1]

SYNOPSIS

This paper is the continuation of the work of P. M. Noble reported at the annual meeting of the Society in 1931.[2] Tests were made on concrete mixes containing sand-gravel aggregate, soft limestone, sandstone, flint gravel, crushed flint aggregate, and a dense limestone. The water-cement ratios varied from 0.6 to 0.9 with slumps from 0 to 8.5 in. The modulus of elasticity and Poisson's ratio were determined on 165 6 by 12-in. cylinders in a moist condition at ages of 14, 28, 56, 112, 380 and 1480 days. Tests were also made on the same specimens at the age of 360 and 1460 days in a dry condition.

Tests show that the modulus of elasticity of plain concrete at the end of 4 yr. is about the same as at the age of 14 days. Poisson's ratio for the mixes studied indicate a rapid increase for the first 56 days and then a gradual decrease until at the age of 4 yr. the ratio was in most cases equal to or less than Poisson's ratio at 14 days.

Sand-gravel aggregate concrete failed to increase in strength, the strength at the age of 4 yr. was in many cases equal to or less than at the age of 28 days.

Sand-gravel aggregate concrete specimens when tested wet gave a lower modulus of elasticity than when the specimens were tested dry. The opposite was true for coarse aggregate specimens. Aggregates have an important influence on the elastic properties of the resulting concrete.

INTRODUCTION

It was the object of this study to determine whether the modulus of elasticity varies as the water-cement ratio, type of aggregate, and age of the concrete. This study covered tests over a period of 4 yr., and involved the taking of over 55,000 measurements for determining the modulus of elasticity and Poisson's ratio on 165—6 by 12-in. concrete cylinders of various mixes, water-cement ratios, and aggregates.

This work was conducted by the Engineering Experiment Station of Kansas State College, under the direction of Prof. C. H. Scholer. The specimens were molded and a preliminary report including tests at the ages of 14, 28, 56 and 112 days was made by P. M. Noble,[2] formerly In-

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[1] Assistant Professor of Applied Mechanics, Kansas State College of Agriculture and Applied Science, Manhattan, Kans.

[2] P. M. Noble, "The Effect of Aggregate and Other Variables on the Elastic Properties of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 399 (1931); also *Bulletin No. 29*, Engineering Experiment Station, Kansas State College of Agriculture and Applied Science, Manhattan, Kans. (1931).

Geo. L. Smith

structor of Applied Mechanics at Kansas State College, and now Field Engineer, Bureau of Reclamation, Yuma, Ariz.

MATERIALS

Cement.—The cement was a brand commonly used in Kansas which met the standard specifications on cement. Sufficient cement was secured at one time for the entire project.

Sand-Gravel Aggregate.—This material is a siliceous material of glacial origin which is commonly called total-aggregate. The material as secured from the field has a fineness modulus of 3.50. The material was screened into three sizes and recombined to give fineness moduli varying from 3.5 to 4.4. The mix was varied from 1:3 to 1:5 by volume, and the water-cement ratios from 0.6 to 0.9.

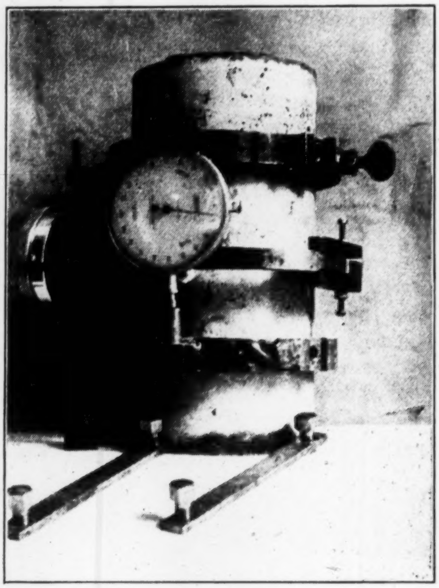

FIG. 1.—Axial and Radial Strainometers on Specimen.

Coarse Aggregate.—Five coarse aggregate materials were used which include a soft local limestone, a gray limestone of fair quality (suitable for concrete pavements), a dense sandstone, a flint aggregate which is a by-product of the zinc and lead mines in southeastern Kansas, and a flint gravel secured from alluvial deposits in Cowley County, Kans.

The coarse aggregate was graded to have a fineness modulus of 7.30 and the fine aggregate used with this material had a fineness modulus of 2.75. The water-cement ratios varied from 0.6 to 0.9 and the slumps varied from 0 to 8 in. The mix was $1:1\frac{3}{4}:3\frac{1}{2}$ by volume. The coarse aggregate was in a saturated and surface-dry condition before mixing. All specimens were mixed, molded and cured according to the Society's Standard Methods of Making Compression Tests of Concrete (C 39 – 33)[1] with the exception that a batch of sufficient size for one series of specimens (five 6 by 12-in. cylinders, and three 3 by 6-in. cylinders) was mixed in a concrete mixer for 2 min.

EQUIPMENT

The radial and axial strainometers used were similar to those used by H. J. Gilkey[2] of Iowa State College, and Stanton Walker[3] at the Structural Materials Research Laboratory.

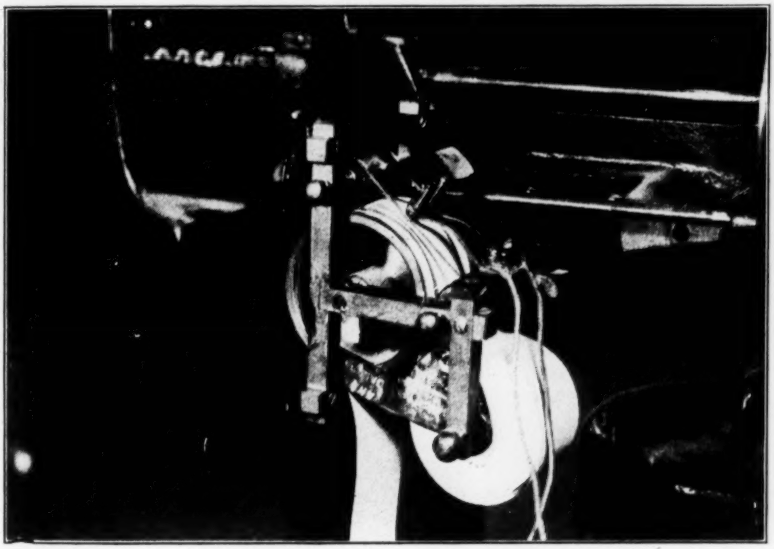

Fig. 2.—Autographic Recording Device; Used for Continuous Loading.

In Fig. 1 is shown the axial and radial strainometers on a specimen ready for testing. These instruments were described by P. M. Noble in the preliminary report.[4] Noble also built an automatic recording device shown in Fig. 2, which greatly facilitated the work in the reading of the loads. The bronze drum is geared to the mechanism which drives the poise of the testing machine. Rubber bands hold a strip of adding machine paper on the drum, the paper traveling as the poise is moved.

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[1] 1933 Book of A.S.T.M. Standards, Part II, p. 230.

[2] Discussion by H. J. Gilkey of paper by C. H. Scholer on "Band Type Circumferential Extensometer for Poisson's Ratio Determinations," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 632 (1930).

[3] Stanton Walker, "Modulus of Elasticity of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. XIX, Part II, p. 510 (1919).

[4] *Loc. cit.*

AGE AND CONDITION AT TIME OF TESTS

The specimens were kept in a moist room maintained at 70 F. until after the specimens were tested at 112 days. After this age the specimens were stored in the laboratory where the maximum temperature did not exceed 100 F., and the minimum relative humidity was 30. These conditions were for short infrequent intervals of time. The average temperature was 70 F. in the winter, and 85 to 90 F. in the summer.

At the age of 365 days the specimens were tested in a dry condition as found in the laboratory, after which specimens were placed in the moist room until the specimens failed to take up any additional moisture, which required 21 days in the moist room. The specimens were then tested in a moist condition. The specimens were then placed in the laboratory air and then tested first dry, then wet, at the ages of 1460 and 1481 days, respectively.

Tests at the different ages were made on the same specimens. Noble found that repeated loadings did not materially affect the slope of the curve. None of the specimens were stressed beyond the elastic limit of the concrete.

METHODS OF TESTING

A typical run involved using the apparatus on the specimen as shown in Fig. 1, and placing the specimen in a 100,000-lb. testing machine. With the poise and each of the dials set at zero, the two buttons were pressed at the proper time making contacts, burning holes in the paper on the automatic recording device, shown in Fig. 2. A continuous load was then applied at the rate of about 280 lb. per sq. in. per min. until a load of 1200 lb. per sq. in. was reached. A mirror was used to facilitate the reading of the radial dial so that one observer could watch both dials at the same time. The operator ran the machine, keeping the poise balanced, while the observer pressed one button when the axial dial recorded a deformation of 0.0005 in. and the second button when the radial dial showed a movement of 0.0001 in. Federal dials were used which could be read directly to 0.0001 in. The dials were checked against Johannsen-Ford gage blocks and were found to be accurate for the range used in making the tests. After reaching a load of 1200 lb. per sq. in. the load was released and the record for the specimen was taken off the automatic recording device. The load was then reapplied to the same specimen and the procedure outlined above repeated. Usually two runs were made on the same specimen, and three specimens were tested of the same mixture, making the results the average of six tests. By using a transparent celluloid scale graduated in unit stress it was possible to interpret the distance between the holes burnt in the recording paper, in terms of stress for each deformation. On the average, ten readings were taken for the axial stress and seven readings for the radial stress for each run.

DETERMINATION OF MODULUS OF ELASTICITY

It is commonly accepted practice to determine the modulus of elasticity of concrete by the secant method at approximately 25 per cent of the compressive strength of the concrete. Such a procedure was followed in this paper. The values given, however, hold true in most cases up to a strength of 1000 lb. per sq. in. or more, which is about 50 per cent of the ultimate strength of the weaker concrete mixtures.

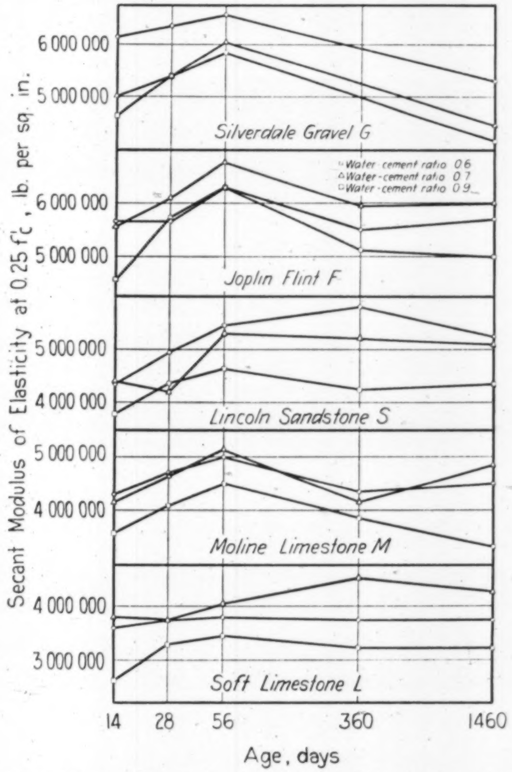

FIG. 3.—Modulus of Elasticity for Various Water-Cement Ratios.
Concrete cylinders 1 : $1\frac{3}{4}$: $3\frac{1}{2}$ by volume. Tested wet at ages of 14, 28, 56 and 112 days, and dry at 360 and 1460 days.

Effect of Water-Cement Ratio on Modulus of Elasticity:

Sand-Crushed Stone Aggregate Concrete.—A study of the modulus of elasticity of concrete by other investigators indicates that the modulus varies with the water-cement ratio. However, they qualify their statement by saying that the modulus does not increase in the same proportion as the water-cement ratio decreases.

Figure 3 shows the modulus of elasticity of concrete cylinders 1 : $1\frac{3}{4}$: $3\frac{1}{2}$ by volume for various water-cement ratios and aggregates at ages varying from 14 days to four years. For a water-cement ratio of 0.6, 0.7 and 0.9 the slumps were approximately 0, 1.50, and 7.0 in. respectively (see Table I).

The flint gravel, G, and sandstone aggregate, S, shows definitely a higher modulus at every age for a water-cement ratio of 0.6 than for 0.7, or 0.9. Soft limestone, L, and flint aggregate, F, showed a definitely higher modulus with a water-cement ratio of 0.7 than for a water-cement ratio of 0.6 or 0.9 (Fig. 3).

TABLE I.—SUMMARY OF DATA ON SPECIMENS.

Batch	Fineness Modulus	Mix by Volume	Water-cement Ratio	Slump, in.	Cement Factor, bbl. per cu. yd.	Modulus of Elasticity, 1000 lb. per sq. in.							
						Wet, 14 days	Wet, 28 days	Wet, 56 days	Wet, 112 days	Dry, 360 days	Wet, 380 days	Dry, 1460 days	Wet, 1480 days
No. 137	4.1	1:3	0.7	7.50	1.85	3268	3559	3897	3873	3608	3562	3445	3208
No. 146	4.1	1:4	0.6	0.25	1.48	3363	4027	4278	4294	....	....	3492	3373
No. 147	4.1	1:4	0.7	1.00	1.52	3509	3906	3950	3970	3608	3485	3223	3124
No. 148	4.1	1:4	0.8	4.75	1.48	3212	3477	3970	3841	....	2987	2601	2705
No. 149	4.1	1:4	0.9	7.50	1.47	2569	3181	3402	3567	....	2463	2744	2664
No. 157	4.1	1:5	0.7	0.00	1.20	2307	3030	3162	3108	....	....	2459	2583
No. 437	4.4	1:3	0.7	8.50	1.89	3339	3717	3973	3952	....	....	3318	3237
No. 447	4.4	1:4	0.7	1.50	1.51	3846	3999	4324	4256	....	3586	3319	3219
No. 457	4.4	1:5	0.7	0.25	1.22	3245	3652	4071	4064	....	3355	3115	3403
No. 537	3.5	1:3	0.7	3.00	1.89	3540	3538	3969	3970	3339	3181	3131	3098
No. 546	3.5	1:4	0.6	0.25	1.45	3175	3357	3832	3678	....	2504	2988	2958
No. 547	3.5	1:4	0.7	0.25	1.46	3350	3458	3505	3775	2973	....	2511	2430
No. 548	3.5	1:4	0.8	1.25	1.51	3082	3382	3499	3736	....	....	2999	2718
No. 549	3.5	1:4	0.9	7.50	1.42	2326	2608	2756	2685	....	2027	2155	2152
No. 837	3.8	1:3	0.7	5.75	1.90	3328	3577	3881	4199	3368	3160	2921	2690
No. 847	3.8	1:4	0.7	0.50	1.56	3322	3639	3899	3882	3372	3123	3078	3172
No. 857	3.8	1:5	0.7	0.00	1.23	1934	2292	2587	2394	....	....	2163	2240
No. 1457	4.1	1:4.5	0.7	0.00	1.45	3259	2991	3473	3404	....	2036	2250	2460
No. 4457	4.4	1:4.5	0.7	0.50	1.38	3328	....	4050	3953	....	3288	3380	3218
No. 5357	3.5	1:3.5	0.7	1.00	1.70	3521	3894	3940	3868	....	2963	2979	2925
No. 8357	3.8	1:3.5	0.7	1.25	1.70	3307	3619	3984	4158	....	3285	3133	3098
Q537[a]	3.5	1:3	0.7	0.25	1.85	2626	3864	4144	4719	4183	3835	3031	3480
C (neat cement)		....	0.4	....	....	3221	3365	3649	....	....	....	1830	1731
G6 Silverdale gravel		$1:1\frac{3}{4}:3\frac{1}{2}$	0.6	0.50	1.45	6142	6345	6531	....	....	....	5288	5744
G7			0.7	2.25	1.45	4959	5378	6035	....	....	....	4472	5293
G9			0.9	7.00	1.42	4652	5396	5868	....	....	....	4147	4671
F6 Joplin flint			0.6	0.00	1.45	5672	5694	6296	....	5505	4700	5720	5670
F7			0.7	1.50	1.47	5564	6090	6764	....	5948	5195	5997	6180
F9			0.9	7.00	1.44	4537	5699	6296	....	5130	5190	5028	5943
L6 Local limestone			0.6	0.00	1.46	3619	3743	3806	....	3743	3760	3726	3569
L7			0.7	1.50	1.53	3796	3781	4054	....	4478	3772	4260	3946
L9			0.9	7.50	1.51	2637	3328	3441	....	3225	2745	3202	3420
M6 Moline limestone			0.6	0.25	1.57	4292	4721	4984	....	4335	....	4500	4520
M7			0.7	1.75	1.58	4154	4610	5133	....	4160	....	4865	5200
M9			0.9	8.00	1.54	3597	4070	4492	....	3858	....	3360	4320
S6 Lincoln sandstone			0.6	0.25	1.52	4364	4895	5369	....	5720	5482	5210	5191
S7			0.7	1.25	1.50	4372	4164	5273	....	5180	5433	5060	5273
S9			0.9	6.00	1.48	3755	4306	4603	....	4199	4044	4329	4393

[a] Early-strength cement.

The aggregate contained in the concrete plays a very important part in the elastic properties of the concrete. Flint aggregate, F, gave a concrete that had a much higher modulus of elasticity than did soft limestone, L. Two aggregates of similar composition such as the flint aggregate, F, and the flint gravel, G, vary in the modulus of elasticity of the resulting concrete. The strength of the concrete containing angular pieces of flint had a higher increase in strength with age than did the flint gravel which had smooth rounded corners (see Table II).

Concrete containing coarse aggregate in most cases had the highest modulus of elasticity at the age of 56 days. The modulus of elasticity of the concrete begins to decrease until at the age of 4 yr. some of the mixtures have decreased as much as 30 per cent from that at 56 days, or a decrease of over 20 per cent from that of 28 days.

Sand-Gravel Aggregate Concrete.—Material such as the Platte River gravel found in Nebraska and western part of Iowa and the sand-gravel aggregate deposits in Kansas, when made into concrete, show desirable qualities. For reinforced concrete work the sand-gravel aggregate is very desirable since because of the small size (maximum $\frac{3}{4}$ in.) it is easy to place, the mix is more workable with a smaller amount of water, and there is not a wide variation in the modulus of elasticity of the concrete for various water-cement ratios.

TABLE II.—SUMMARY OF STRENGTH AND POISSON'S RATIO ON SPECIMENS TESTED.

Batch	3 by 6-in. Cylinders, Compressive Strength,[a] 28 days, lb. per sq. in.	6 by 12-in. Cylinders, Compressive Strength,[b] 1480 days, lb. per sq. in.	Poisson's Ratio							
			Wet, 14 days	Wet, 28 days	Wet, 56 days	Wet, 112 days	Dry, 360 days	Wet, 380 days	Dry, 1460 days	Wet, 1480 days
No. 137	3925	3877	0.158	0.200	0.204	0.213	0.168	0.170	0.164	0.161
No. 146	1937	3400	0.150	0.190	0.184	0.207	.....	.....	0.147	0.164
No. 147	2948	3538	0.180	0.188	0.187	0.206	0.171	0.165	0.154	0.181
No. 148	3354	3044	0.168	0.176	0.185	0.219	.....	0.183	0.146	0.160
No. 149	2982	2717	0.165	.....	0.176	0.192	.....	0.200	0.195	0.184
No. 157	2228	1990	0.192	0.178	0.208	0.214	.....	.....	0.191	0.223
No. 437	3982	3869	0.158	0.170	0.205	0.198	.....	.....	0.130	0.187
No. 447	4080	3727	0.192	0.140	0.205	0.194	.....	0.162	0.147	0.144
No. 457	3406	3104	0.177	0.178	0.205	0.189	.....	0.208	0.146	0.192
No. 537	3459	3131	0.177	0.204	0.172	0.200	0.171	0.143	0.146	0.152
No. 546	2933	2249	0.217	0.180	0.247	0.204	.....	0.174	0.166	0.188
No. 547	3060	2717	0.189	0.206	.....	0.195	0.160	.....	0.150	0.185
No. 548	2933	2991	0.159	0.169	0.198	0.192	.....	.....	0.147	0.176
No. 549	2604	2408	0.198	.....	0.212	0.198	.....	0.211	0.173	0.198
No. 837	3768	3720	0.155	0.271	.....	0.161	0.161	0.160	0.160	0.161
No. 847	3309	3129	0.194	0.191	0.225	0.162	0.140	0.164	0.153	0.189
No. 857	2006	1785	0.400	0.262	0.358	0.303	.....	.....	0.326	0.290
No. 1457	2581	2276	0.180	0.168	0.214	0.255	.....	0.252	0.207	0.248
No. 4457	3038	3341	0.130	.....	0.211	0.193	.....	0.196	0.173	0.160
No. 5357	3722	3341	0.208	0.189	0.158	0.197	.....	0.184	0.153	0.176
No. 8357	3763	3749	0.169	0.136	0.185	0.214	.....	0.181	0.136	0.159
Q537	2202	3844	0.184	0.182	0.171	0.205	0.192	0.160	0.213	0.146
C	7272	4600	0.268	0.243	0.282	.....	.....	.....	0.124	0.097
G6	4145	5385	0.137	0.148	0.161	.....	.....	.....	0.085	0.112
G7	3087	4155	0.166	0.180	0.166	.....	.....	.....	0.126	0.135
G9	2896	3230	0.219	0.192	0.155	.....	.....	.....	0.120	0.137
F6	4188	5400	0.167	0.169	0.201	.....	0.128	0.135	0.132	0.167
F7	3056	5288	0.163	0.167	0.166	.....	0.135	0.085	0.131	0.189
F9	2436	3728	0.118	0.224	0.167	.....	0.165	0.100	0.098	0.133
L6	3740	4127	0.235	0.192	0.210	.....	0.175	0.188	0.221	0.171
L7	4060	4687	0.178	0.202	0.217	.....	0.205	0.187	0.191	0.171
L9	2090	3170	0.177	0.223	0.219	.....	0.145	0.195	0.200	0.186
M6	4647	5912	0.230	0.197	0.177	.....	0.238	.....	0.167	0.184
M7	4405	6513	0.166	0.176	0.209	.....	0.169	.....	0.124	0.161
M9	1938	4197	0.190	0.198	0.192	.....	0.200	.....	0.135	0.183
S6	4051	5639	0.128	0.180	0.188	.....	0.141	0.174	0.143	0.118
S7	3940	5251	0.159	0.175	0.205	.....	0.132	0.155	0.166	0.107
S9	2438	3871	0.178	0.186	0.164	.....	0.147	0.120	0.138	0.117

[a] Each value is the average of 3 specimens.
[b] Each value is the average of 3 to 5 specimens.

As shown in Fig. 4, for a fineness modulus of 3.5 (mix 1:4 by volume) there is a wide variation in modulus of elasticity between a water-cement ratio of 0.8 and 0.9.

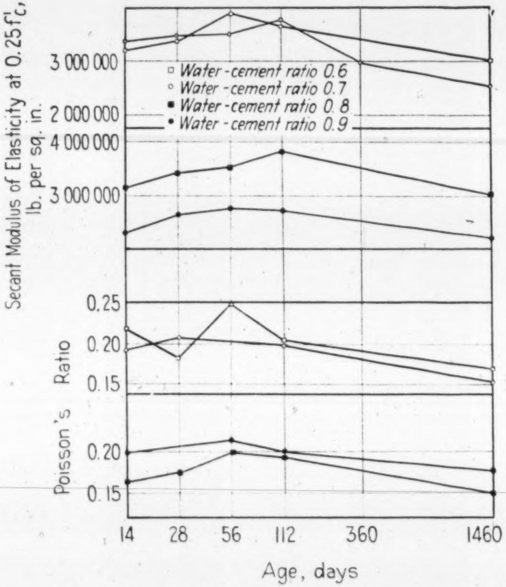

FIG. 4.—Modulus of Elasticity and Poisson's Ratio for Various Water-Cement Ratios.

Concrete cylinder 1:4 by volume. Fineness modulus 3.5.

Tested wet at ages of 14, 28, 56 and 112 days, and dry at 360 and 1460 days.

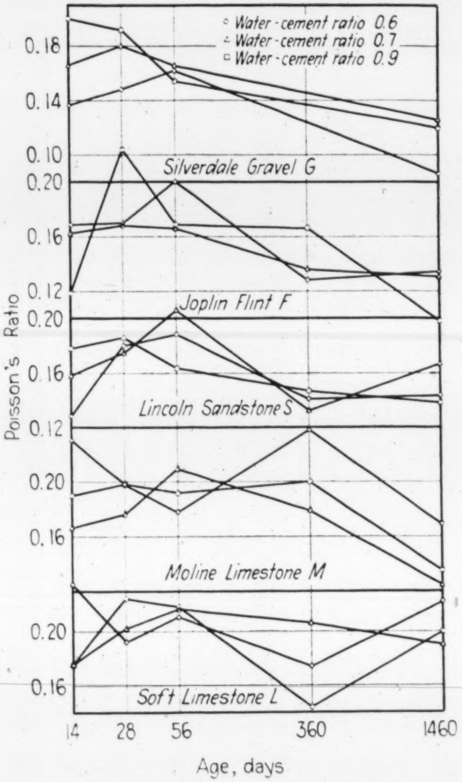

FIG. 5.—Poisson's Ratio for Various $\frac{w}{c}$ Ratios.

Concrete cylinder $1:1\frac{3}{4}:3\frac{1}{2}$ by volume.

Tested wet at ages of 14, 28, 56 and 112 days, and dry at 360 and 1460 days.

For a better graded aggregate (fineness modulus of 4.1) mix 1:4 by volume, the variation in modulus of elasticity was less than 20 per cent for water-cement ratios of 0.6 and 0.9.

In most cases the maximum value for modulus of elasticity is found at the age of 56 days.

In practically every case the strength of the sand-gravel aggregate concrete decreased slightly with age up to the time of the final tests, which was at the age of 4 yr. Concrete specimens made of sand-gravel aggregate showed a decrease of 3 to 20 per cent in the modulus of elasticity over a 4-yr. period, depending upon the richness of the mix.

A comparison of the compression tests on 3 by 6-in. cylinders at the age of 28 days and the compression tests on 6 by 12-in. cylinders at the age of 4 yr. shows that there is a slight tendency for the sand-gravel aggregate concrete to decrease in strength with age, instead of increasing as did the concrete containing the coarse aggregate. Although the decrease is slight it is significant since each of the compression values in the table represents the average of three specimens. Nor can the difference be attributed to the tests being made on 3 by 6-in. cylinders at 28 days and on 6 by 12-in. cylinders at 4 yr. Engineers have found from actual tests that the difference in compressive strength when testing in the above sizes is neglible. For coarse aggregate specimens the increase with age was very noticeable, increasing as much as 70 per cent in the "F" series (see Table II), with an average of about 20 to 25 per cent increase for all coarse aggregate specimens.

POISSON'S RATIO

As reported by other authors there is no definite relationship between the compressive strength of the concrete and Poisson's ratio. Perhaps the most interesting fact brought out by these tests is that the Poisson's ratio first increases up to the age of 56 to 112 days for sand-gravel aggregate concrete (see Table II), after which the ratio decreases until at the ages of 1 and 4 yr., Poisson's ratio for many of these materials and mixes is less than at the age of 14 days. For sand-gravel aggregate concrete this decrease in Poisson's ratio cannot be attributed to the strength of the concrete since it decreased slightly with age. For coarse aggregate concrete the strength of the resulting concrete increased with age and since Poisson's ratio decreased in most cases from that at the age of 28 days (see Fig. 5), this indicates that the aggregate in the concrete has been thoroughly cemented together, resisting lateral deformation with the application of load. When the specimens were tested wet, Poisson's ratio increased in most instances.

DISCUSSION OF TEST DATA

It will be noticed that the results contained in this report, for the most part, conflict with the data secured by Davis and Troxell,[1] Walker,[2] and others on concrete specimens made and tested 6 to 17 yr. ago. Considerable information has been secured on concrete during the past 10 yr.; apparatus has been perfected which permits greater accuracy in measuring certain properties; equipment has been secured which permits a closer control on the curing and testing of specimens. All of these factors have assisted in obtaining a better understanding of the behavior of concrete.

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[1] R. E. Davis and G. E. Troxell, "Modulus of Elasticity and Poisson's Ratio for Concrete, and the Influence of Age and Other Factors upon These Values," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 678 (1929).

[2] *Loc. cit.*

The following explanation may account for the different results secured by the author and others:

1. The refinement of equipment in curing, testing, and measuring loads and deformations has been greatly improved over that of previous tests.

2. The finer grinding of cement and the better control in the manufacture of cement has aided in a better and more uniform product, the characteristics of which may not be quite the same as the cements of 10 yr. ago.

3. The results contained in this paper cover tests over a 4-yr. period which is a longer study on the subject than had been made heretofore on plain concrete.

4. The studying of aggregates has been somewhat neglected, perhaps because of the difficulty of measuring the qualities such as Poisson's ratio, modulus of elasticity, volume change, and the effect of gradation on the resulting volume change.

A few investigators have been gathering data on the aggregates themselves, but as yet no definite procedure has been set up for the determination of the above properties. Knowing the individual properties of the aggregates entering into the concrete and the characteristics of the cement, it may be possible to account for some of the conflicting data published. It is the author's opinion that the data published in the past have perhaps been fairly accurate, but the materials entering into the specimens have been enough different to give conflicting results when compared with data from specimens made in a different section of the country.

CONCLUSIONS

1. In studying the characteristics of concrete mixtures, tests should be made over a period of time up to 4 or 5 yr.

2. Compression tests on sand-gravel aggregate concrete indicate that there is a slight decrease in strength (when compared with 28-day strengths) after 4 yr. in laboratory air.

3. The modulus of elasticity of concrete at the age of 4 yr. is practically the same as that at the age of 14 days.

4. Poisson's ratio increases rapidly for most concretes up to an age of 56 days, the ratio then decreases in most cases until it is equal to or less than the ratio at the age of 14 days.

5. Sand-gravel aggregate concrete specimens when tested wet give a lower modulus of elasticity than when tested dry; the opposite is true for coarse aggregate concrete specimens.

6. The aggregate in concrete plays a very important part so far as the strength and elastic properties of the concrete are concerned.

7. A definite method is needed to determine the modulus of elasticity, volume change, and Poisson's ratio on the individual aggregates entering into the concrete mixtures.

8. Compression tests on concrete samples should be made at ages up to at least 5 yr. to determine definitely the trend of the behavior of the concrete which may not be indicated at the ages of 1 or 2 yr.

RESEARCH ON CONCRETE DISINTEGRATION

By H. S. Mattimore[1] and G. A. Rahn[1]

Synopsis

This paper presents the results of a research project undertaken to determine the cause of an abnormal rate of disintegration of concrete structures in northern Pennsylvania.

Forty-five experimental concrete headwalls were constructed in the same location as the original walls which had failed, and these were closely observed and their resistance to exposure recorded. Two different sands, and two different cements with the same coarse aggregate were used; also the cement and aggregate proportions and water-cement ratio were varied.

The inspection record, with the available detail data on material qualities, concrete proportioning, compressive strength, pH determinations on surrounding soils and daily recording thermometer records over the entire exposure period furnishes information rarely, if ever, available on studies of field concrete. The periodic inspections made it possible to record the development of disintegration and to establish a rating on stages of disintegration, which it is hoped will be of value in recording data on similar investigations.

Some of the probable causes for disintegration are discussed in the paper, but what is believed to be of more value is that sufficient data are furnished so these causes can be checked or other conclusions drawn.

Introduction

While the many concrete structures that are giving efficient service over many years have justified confidence in the durability of good concrete, possibly this generally successful use has led to over confidence and the assumption that all concrete materials can be successfully used under all conditions of service and exposure. This subject is now receiving some attention, as is evidenced by recent trends to use special cements for special conditions of service and exposure.

The present investigation was undertaken to try to determine the causes for the disintegration of concrete headwalls in McKean County, Pa., less than one year after construction. The failures were investigated when this disintegration was observed but no acts of omission or commission of good engineering practice or specification evasion could be found. In fact, all available data and an analysis of the hardened concrete definitely determined that all requirements of the specifications had been fulfilled.

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[1] Engineer of Tests, and Research Engineer, respectively, Pennsylvania State Highway Dept., Harrisburg, Pa.

TABLE I.—CONCRETE DURABILITY DATA AND RATINGS ON EXPERIMENTAL HEADWALLS AT $4\frac{1}{2}$ YR. AFTER PLACING.

NOTE.—Stone from same source used in all headwalls.

Headwall	Cement	Sand	Mix	Water, gal. per bag	Water-Cement Ratio, $\frac{w}{c}$	28-day Compressive Strength, lb. per sq. in.	Rating[e] at $4\frac{1}{2}$ yr., per cent
No. 1	A	C	$1:2:3\frac{1}{2}$	5.22	0.70	3890	60
No. 2			$1:2:3\frac{1}{2}$	5.72	0.76	4138	80
No. 3[a]			$1:2:3\frac{1}{2}$	5.72	0.76	2759	70
No. 4[b]			$1:2:3\frac{1}{2}$	5.72	0.76	3795	50
No. 5			$1:2:3\frac{1}{2}$	5.72	0.76	3112	50
No. 6			$1:2:3\frac{1}{2}$	5.72	0.76	2936	60
No. 7			$1:2:3\frac{1}{2}$	7.05	0.94	2974	0
No. 8			$1:2:3\frac{1}{2}$	6.55	0.88	3537	30
No. 9			$1:2\frac{1}{2}:5$	7.15	0.96	4180	80
No. 10			$1:2\frac{1}{2}:5$	7.40	0.99	3289	85
No. 11[a]			$1:2\frac{1}{2}:5$	7.40	0.99	3040	55
No. 12			$1:2\frac{1}{2}:5$	7.40	0.99	2656	60
No. 13			$1:2\frac{1}{2}:5$	7.90	1.04	2057	30
No. 13-A			$1:2\frac{1}{2}:5$	9.40	1.25	1736	40
No. 14			$1:1\frac{1}{2}:3$	4.79	0.64	4357	60
No. 15			$1:1\frac{1}{2}:3$	5.04	0.67	4226	50
No. 16			$1:1\frac{1}{2}:3$	5.79	0.77	3749	10
No. 17		D	$1:2:3\frac{1}{2}$	5.37	0.72	4669	80
No. 18			$1:2:3\frac{1}{2}$	5.97	0.80	3572	80
No. 19			$1:2:3\frac{1}{2}$	6.72	0.85	2476	80
No. 20			$1:2\frac{1}{2}:5$	7.15	0.95	4386	70
No. 21			$1:2\frac{1}{2}:5$	7.90	1.04	2900	80
No. 22			$1:2\frac{1}{2}:5$	9.40	1.25	2016	80
No. 23			$1:1\frac{1}{2}:3$	5.14	0.69	4577	70
No. 24			$1:1\frac{1}{2}:3$	5.34	0.71	4760	80
No. 25			$1:1\frac{1}{2}:3$	5.79	0.77	4353	60
No. 26	B	C	$1:2:3\frac{1}{2}$	5.55	0.74	3784	70
No. 27			$1:2:3\frac{1}{2}$	5.72	0.76	3678	80
No. 28			$1:2:3\frac{1}{2}$	6.47	0.86	3695	80
No. 29			$1:2\frac{1}{2}:5$	6.90	0.92	2943	85
No. 30			$1:2\frac{1}{2}:5$	7.40	0.99	2221	90
No. 31			$1:2\frac{1}{2}:5$	9.40	1.25	1577	90
No. 32			$1:1\frac{1}{2}:3$	4.79	0.64	3926	70
No. 33			$1:1\frac{1}{2}:3$	4.99	0.67	3678	70
No. 34			$1:1\frac{1}{2}:3$	5.79	0.77	3745	80
No. 35		D	$1:2:3\frac{1}{2}$	5.39	0.72	3926	93
No. 36			$1:2:3\frac{1}{2}$	5.72	0.76	3749	80
No. 37			$1:2:3\frac{1}{2}$	6.72	0.90	2044	85
No. 38[c]	A	C	$1:2:3\frac{1}{2}$	5.72	0.76	3275	60
No. 39	B	D	$1:2\frac{1}{2}:5$	7.15	0.96	2391	90
No. 40			$1:2\frac{1}{2}:5$	7.90	1.04	2334	90
No. 41			$1:2\frac{1}{2}:5$	9.65	1.29	1995	90
No. 42			$1:1\frac{1}{2}:3$	5.04	0.67	4499	80
No. 43			$1:1\frac{1}{2}:3$	5.44	0.73	4513	80
No. 44			$1:1\frac{1}{2}:3$	5.79	0.79	3473	80
No. 45[d]	A	C	$1:2:3\frac{1}{2}$	5.72	0.76	3749	..

[a] Precast.
[b] Waterproofed.
[c] Harrisburg City water.
[d] Buried under ground.
[e] Rating in per cent of intact structure.

Since no causes for this disintegration could be found by this method of investigation, it was decided to construct a series of headwalls similar to those that had failed and to subject them to similar conditions of exposure at the same site. Forty-five concrete headwalls, 36 by 12 by 36 in. were constructed with 2 ft. below the surface of the ground, allowing 1 ft. of the concrete to be exposed to the atmosphere. Two different cements, designated cement A and cement B, and two different sands designated sand C and sand D were used in the concrete in different walls, with variations in mix proportions and water-cement ratios. An analysis of the portland cements and physical properties of the sands are given in Tables III and IV.

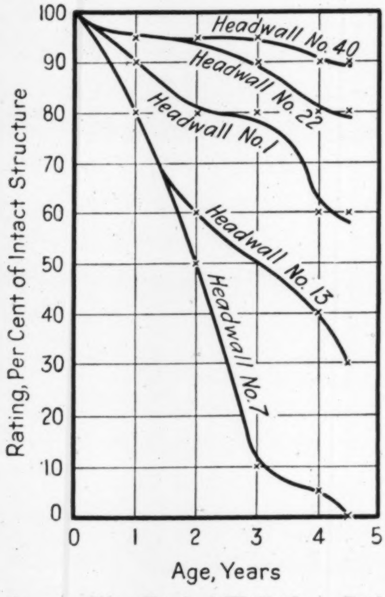


FIG. 1.—Ratings on Five Typical Walls Over Period of $4\frac{1}{2}$ yr.

The same coarse aggregate was used throughout. Figure 6 shows a record of the variation in temperature during the calendar year 1934 which gives an idea of the climatic conditions encountered. Table V shows the pH values of the soils for each headwall location which indicates that the soils are practically neutral.

COMPARISON OF DURABILITY

The rating of the walls at the inspection $4\frac{1}{2}$ yr. after placing is recorded in Table I. This record shows a distinct difference in the structural rating of the walls, varying from 0 per cent to 93 per cent. It will be noted that no walls are rated as 100 per cent as it was found difficult to differentiate between walls in the 95-per-cent rating and those in the 100-per-cent

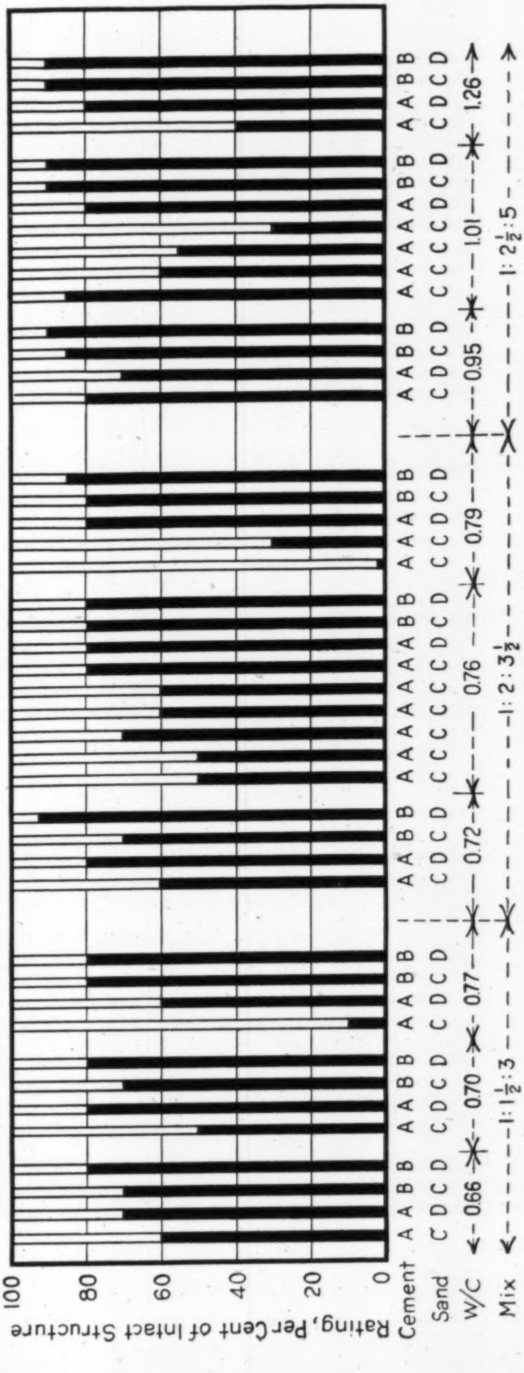
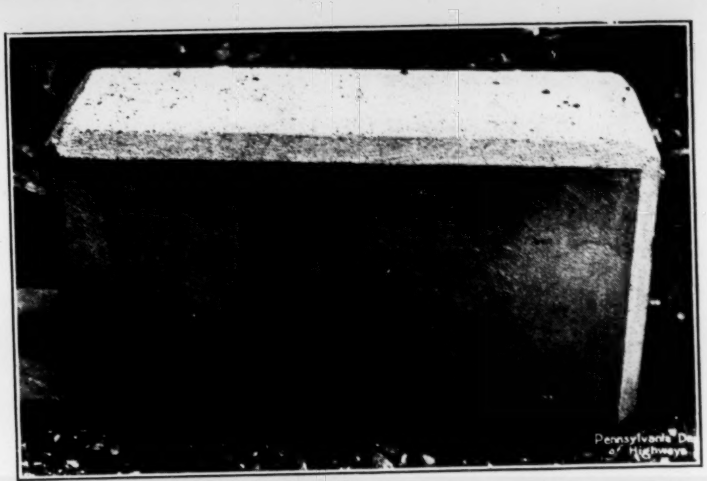
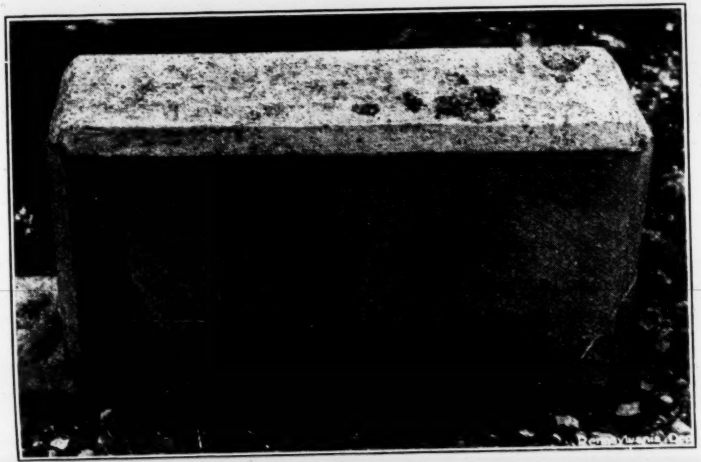


FIG. 2.—Concrete Durability Ratings on Experimental Headwalls at 4½ yr.

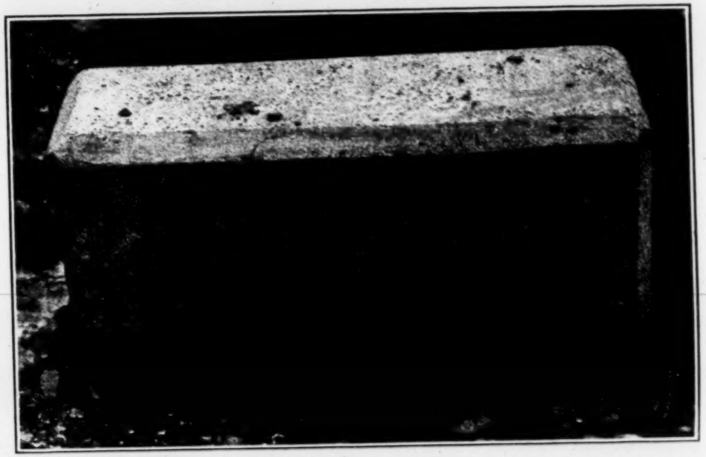
Ratings A and B—100 and 95 per cent of design strength.
Rating A—*Structure intact.*
Rating B—*Individual cracks discernible with reading glass.* This condition marks the start of disintegration.



Rating C—90 per cent of design strength.
Individual cracks discernible to naked eye. The point when the start of disintegration becomes apparent to the casual observer.

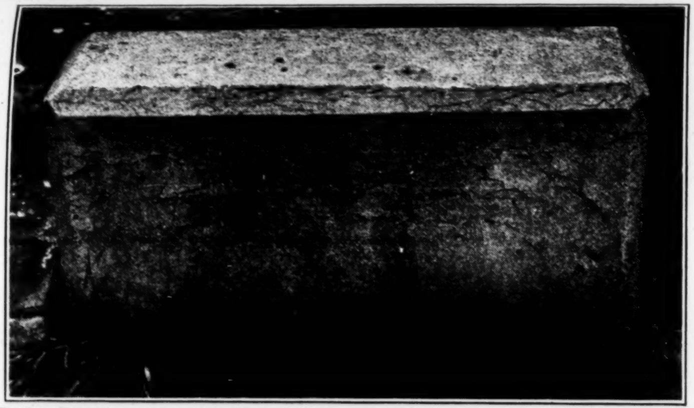


Rating D—80 per cent of design strength.
Connection of cracks apparent to naked eye, crow feet. The next step, disintegration becomes more pronounced. Up to this point while disintegration has progressed, it has not greatly reduced the inherent strength of the structure as the action is still local and has not become general.

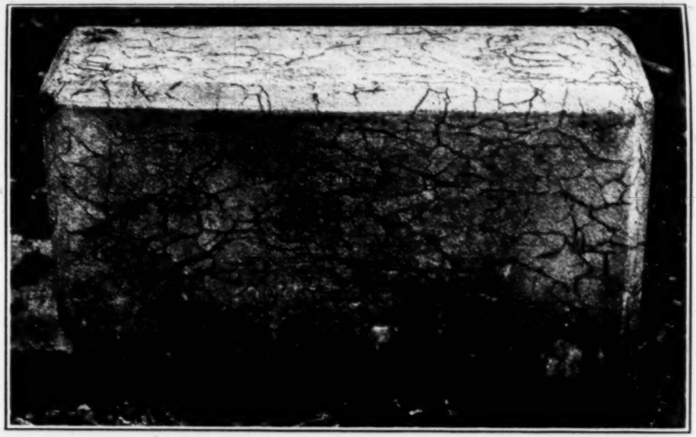


Rating E—60 per cent of design strength.
Connecting of cracks, alligator checks in spots. At this point while the action may still be local it has become concerted and has affected areas, thereby affecting the increment strength.

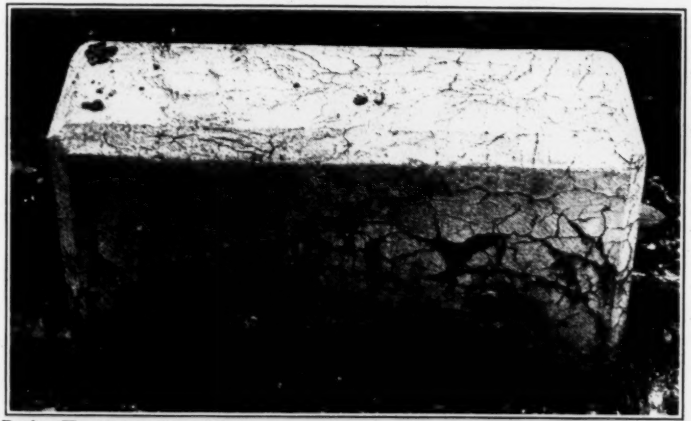
FIG. 3.—Showing a Correlation of Alphabetical and Numerical Ratings.



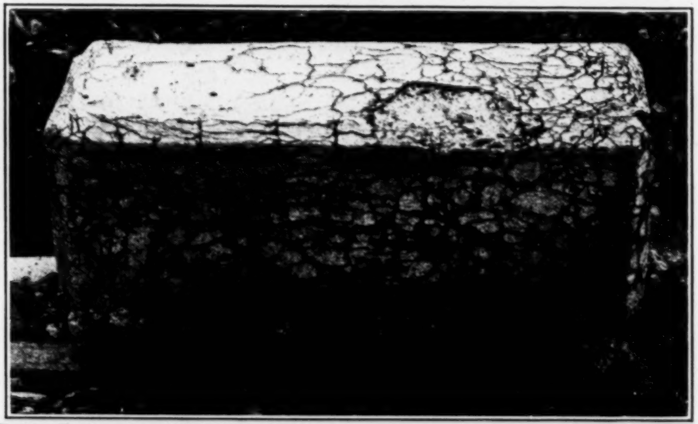
Rating F—50 per cent of design strength.
Alligator checks continuous, cracks tending to open. The entire structure is affected with a further decrease in strength. From this point the disintegration is very rapid and the structure falls an easy prey to thermal stress and the prying action of ice.



Rating G—30 per cent of design strength.
Pronounced widening of cracks—disintegration started. Thermal stresses and ice at work.



Rating H—10 per cent of design strength.
Disintegration progressed to marked degree. While the structure maintains its original shape it is possible to disintegrate same with but very little effort.



Rating I—0 per cent of design strength.
Complete disintegration of structure—sections dislodged and falling from same.

FIG. 4.—Showing a Correlation of Alphabetical and Numerical Ratings.

rating, so that there is a good possibility that at least some of those rated 95 per cent and better are fully intact structurally. Ratings on five typical walls over a period of 4½ yr. are shown in Fig. 1.

An interesting comparison in Table I is the structural rating of individual walls and the 28-day compressive strength on cylinders made from concrete placed in such walls. For instance, walls Nos. 39, 40 and 41 have compressive strengths of 2391, 2334 and 1995 lb. per sq. in., respectively, yet all have structural ratings of 90 per cent, while walls Nos. 7 and 16, with structural ratings of 0 and 10 per cent have compressive strengths of 2974 and 3749 lb. per sq. in., definitely illustrating that compressive strength is not sufficient control to assure durability in concrete.

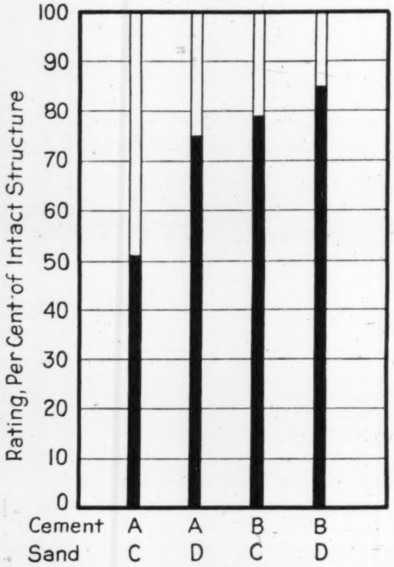


FIG. 5.—Material Comparison, Average of all Mixes and Consistencies at 4½ yr.

A comparison of different mixes classified in accordance with water-cement ratios in Fig. 2 shows that in general the wetter mixes are less durable than the drier ones; also mixes rich in cement show lower structural rating than the leaner mixes. This latter is somewhat perplexing in that usually richer mixes are more dense, therefore, less permeable—factors which should be conducive to good durability. Volume change due to different temperatures of exposed and buried sections of walls, and the particular materials used might be at least partially responsible for failures in the rich mixes.

The photographs reproduced as Figs. 3 and 4 illustrate the rating established for structural conditions. For convenience of field inspection these ratings are given in terms of letters from A to I.

COMPARISON OF MATERIALS

Material combinations are segregated in Fig. 5 plotted from data in Table I and it can be readily observed that mixes where cement B was used have higher structural rating than mixes where cement A was used; also sand D produced more durable concrete than did sand C and further the combination of cement A and sand C resulted in the lowest rating of any combination. A detail examination of the physical tests on cements A and B in Table II indicates little or no difference in quality, judging by the usual requirement of standard specifications. Also a study of Table III does not indicate a major difference in analysis or calculated compounds, that is, if we are only to consider compounds that have come into prom-

TABLE II.—TESTS ON PORTLAND CEMENTS USED IN HEADWALLS.

	CEMENT A		CEMENT B	
Fineness, retained on No. 200 sieve, per cent	8.8		13.5	
Initial set, min.:				
Maximum	100		205	
Minimum	85		180	
Average	91		190	
Final set, min.:				
Maximum	150		295	
Minimum	145		275	
Average	149		284	
Tensil Strength, lb. per sq. in.:	7 DAYS	28 DAYS	7 DAYS	28 DAYS
Maximum	385	...	332	...
Minimum	347	...	323	...
Average	370	467	328	412
Micron sizes:				
30 micron	51.73		49.52	
20 micron	36.65		35.22	
15 micron	31.34		28.91	
10 micron	24.71		23.11	

TABLE III.—ANALYSES OF PORTLAND CEMENTS USED IN HEADWALLS.

	Chemical Analyses, per cent			Calculated Compounds, per cent	
	Cement A	Cement B		Cement A	Cement B
Silica (SiO_2)	20.16	20.92	Tricalcium silicate ($3\,CaO \cdot SiO_2$)—C_3S	45.0	41.1
Iron oxide (Fe_2O_3)	3.36	2.07	Dicalcium silicate ($2\,CaO \cdot SiO_2$)—C_2S	24.0	29.1
Alumina (Al_2O_3)	5.70	5.41	Tricalcium aluminate ($3\,CaO \cdot Al_2O_3$)—C_3A	9.4	10.9
Calcium oxide (CaO)	60.53	60.07	Tetracalcium aluminum ferrate ($4CaO \cdot Fe_2O_3 \cdot Al_2O_3$)—$C_4AF$	10.2	6.3
Magnesia (MgO)	1.38	4.17	Calcium sulfate ($CaSO_4$)	3.1	3.0
Sulphuric anhydride (SO_3)	1.83	1.74	Magnesia	1.4	4.2
Loss on ignition	6.09	5.18	Loss on ignition	6.1	5.2
Insoluble residue	0.20	0.10			
Alkalies Na_2O	0.43	0.24			
Alkalies K_2O	0.57	0.37			

inence in recent specifications. The calculated C_3S and C_3A content compares favorably in both cements. In fact the only difference of any account is in the C_4AF content which has to a large extent been ignored in most specifications that limit the amount of certain compounds. Actually in some locations the reduction of C_3A compound is accomplished at the expense of an increase in the C_4AF content. Again referring to Table III, although the alkali content is very small, there is a noticeable difference in the alkali content of these two cements.

Table IV.—Tests on Sands Used in Headwalls.

	Sand Gradation, per cent	
	Sand C	Sand D
Passing $\frac{3}{8}$-in. sieve	100.0	100.0
Passing No. 4 sieve	95.8	99.0
Passing No. 20 sieve	53.7	66.2
Passing No. 50 sieve	12.1	13.4
Passing No. 100 sieve	2.6	1.0

Sodium Sulfate Soundness of Sands

Sand C loss = 17.3 per cent

Sand D loss = 5.2 per cent

Table V.—pH Values of Soils.

Samples at each headwall location.

Headwall	pH Value	Headwall	pH Value	Headwall	pH Value
No. 1	6.0	No. 15	6.0	No. 31	5.2
No. 2	5.4	No. 17	6.0	No. 32	5.2
No. 3	5.8	No. 18	6.0	No. 33	5.2
No. 4	5.3	No. 19	5.8	No. 34	5.8
No. 5	5.8	No. 20	5.9	No. 35	5.1
No. 6	5.8	No. 21	6.2	No. 36	5.6
No. 7	5.6	No. 22	6.0	No. 37	5.7
No. 8	5.8	No. 23	5.2	No. 38	5.3
No. 9	5.3	No. 24	5.2	No. 39	5.5
No. 10	5.2	No. 25	6.3	No. 40	5.7
No. 11	5.3	No. 26	5.1	No. 41	5.6
No. 12	5.5	No. 27	5.2	No. 42	5.8
No. 13	6.3	No. 28	5.1	No. 43	5.2
No. 13-A	6.6	No. 29	5.0	No. 44	5.6
No. 14	6.3	No. 30	5.2		

Comparing sand C and sand D, concrete walls in which sand D was used generally had higher structural rating than the concrete where sand C was used. This is especially noticeable in combinations of cement A and sand C. The loss in sodium sulfate test (Table IV) of sand C as compared with sand D gives a close check on the difference in durability when used in concrete.

RECOMMENDATIONS

It is suggested that further study be given the C_4AF and alkali content in cements to be used in concrete subject to extreme exposure and rapid alterations of temperature such as these walls were. Successful results

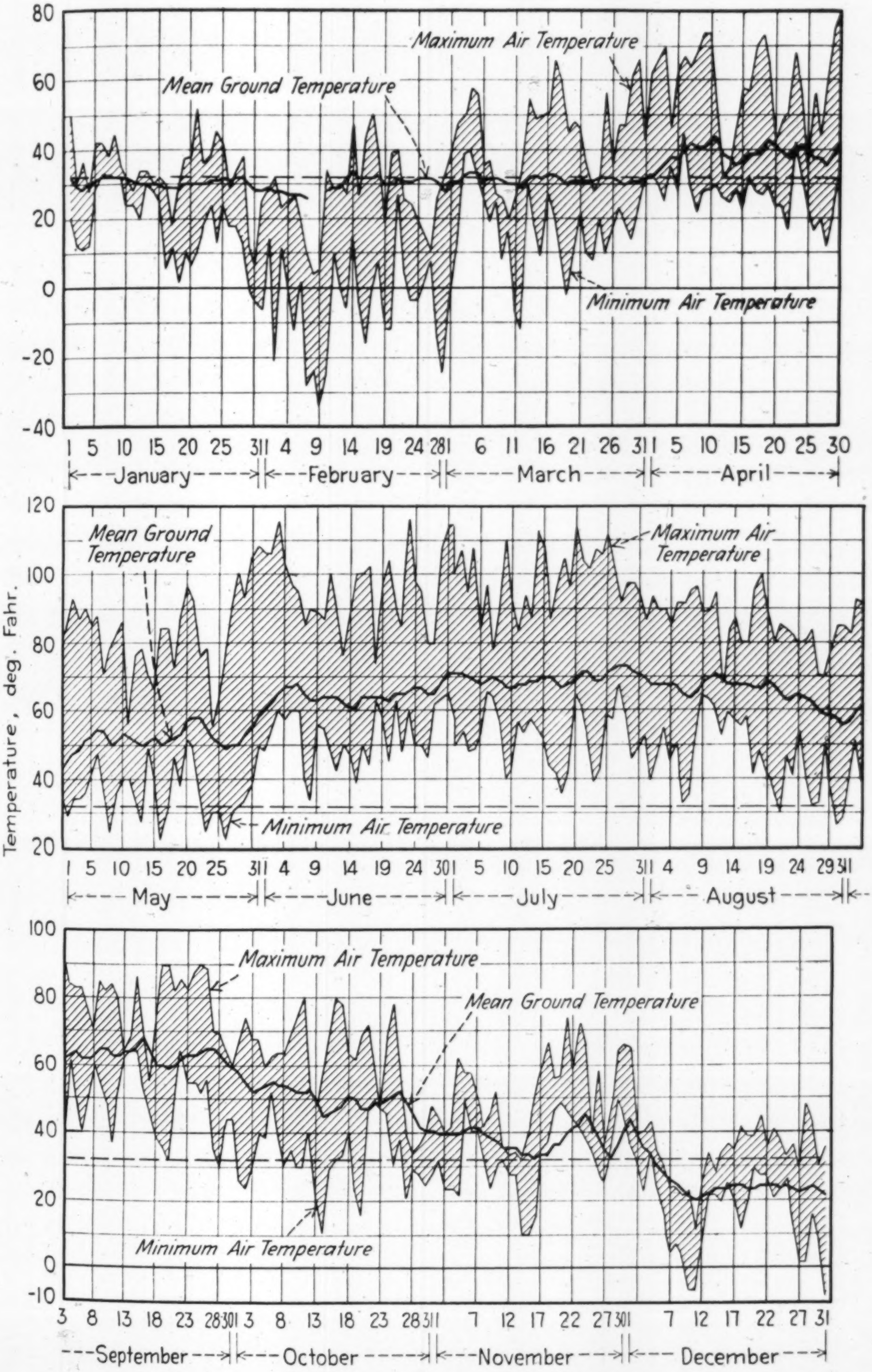


FIG. 6.—Temperature Range and Mean Ground Temperature (1934).

with cement A in other locations indicate that this problem is a special one, probably requiring special cements. Although cement B has produced more durable concrete, the time of exposure has been too limited to indicate whether even this cement will give full efficiency.

The progress of disintegration is of some interest for study, as illustrated in Fig. 1, showing progress of five typical walls.

CONCLUSIONS

Extremes in temperature between exposed section and buried part of wall subject these specimens to high stresses.

Saturated ground conditions with rather frequent changes in temperature produced numerous cycles of freezing and thawing during the 4½ yr. of exposure.

Cement A and sand C are definitely indicated as factors affecting adversely the durability of concrete under this condition of exposure.

Table V giving the pH value of soils in contact with these walls is convincing that deleterious acid or alkali action is not a factor in this disintegration.

DISCUSSION

Mr. F. O. Anderegg.[1]—The photographs shown in Mr. Mattimore's paper give every indication of disintegration resulting from sulfate action, and it is suggested that sulfate determinations of the adjacent soil and of the rainwater falling during the period might well have produced valuable information. A visit to the region in 1930 yielded a series of concrete pavement samples in which a good correlation between age of concrete and sulfoaluminate content was obtained with the aid of a petrographic microscope by my colleague, D. S. Hubbell. The higher alkali content of cement A meant more sulfate held in solution and available for attack after setting. Generally the C_4AF compound has been found helpful in resisting sulfate attack and it is respectfully suggested that Mr. Mattimore look elsewhere for the cause of the trouble.

Mr. C. H. Scholer.[2]—I wish to compliment Mr. Mattimore for having approached the problem of the durability of concrete from the proper angle, that of a field investigation. It has long been my belief that such studies must start where the difficulties are observed, and from this point the problem can perhaps be transferred to the laboratory for investigation. The results of such a preliminary study cannot hope to be more than suggestive of future methods of attack.

In 1928 I reported to the Society the results of a field investigation of this nature started in 1925,[3] together with some laboratory studies carried on simultaneously. These studies have been very helpful in interpreting the significance of defects observed in structures exposed to weather and other elements, and also as a guide in outlining further studies of durability. They have not, however, answered the many questions that arise as to why some concrete is more durable than other concrete of comparable quality as regards strength. I think Mr. Mattimore will find that as time goes on his project will be of increasing value, but that it will probably not explain why the failures occurred.

Anyone interested in the details of the Kansas project started in 1925 can find them in the reference previously cited. The field specimens are still under observation and after ten years, considerable progress in deterioration has been noted on the very lean field mixes. These are all at about the same general level after these years of exposure. The leaner field specimens have failed completely and the good laboratory specimens are still in practically perfect condition. During the first few years there appeared to be more variation between specimens at different locations than is apparent now after ten years of exposure.

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[1] Consulting Specialist on Building Materials, Mapleton Depot, Pa.

[2] Professor of Applied Mechanics, Kansas State College of Agriculture, Road Materials Laboratory, Manhattan, Kans.

[3] C. H. Scholer, "Some Accelerated Freezing-and-Thawing Tests on Concrete," ***Proceedings***, Am. Soc. Testing Mats., Vol. 28, Part II, p. 472 (1928).

The general appearance of the concrete as shown by the photographs given in the paper would lead me to suspect that unsatisfactory aggregates probably played an important part in the failures observed.

The differences in the composition of the cements would hardly be sufficient to explain the results shown.

The differences in the result with identical specimens tend to bring out the difficulty in securing significant results in durability tests unless large numbers of specimens are used. Referring to Figs. 4 and 5, it will be noted that the combination of cement A and sand C in four headwalls of identical composition shows a wider variation (85 to 30 per cent) than the average of the best combination as compared with the worst combination of cement and sand.

I was much interested in noting the weather record which Mr. Mattimore took in connection with this project. Such data are extremely scarce and it is to be hoped that this record will be continued. There is at present no available information as to the probable number of freezing and thawing cycles to which concrete structures will be subjected under actual weather conditions. Mr. Mattimore's data on ground temperatures should be of much assistance in estimating the probable number of cycles per year. Mr. Mattimore's record for 1934 indicates 23 cycles for that year.

MR. H. F. GONNERMAN[1] (*by letter*).—Through the courtesy of Mr. Mattimore, the writer had the opportunity to inspect the headwalls described in the paper. During the inspection, Mr. Rahn and the writer rated the walls independently, according to the authors' established method, and were in fair agreement.

The writer feels, however, that the authors' system of rating the walls is somewhat severe. The rubbed mortar finish, as applied to the walls, is known to be conducive to surface checking. These minute surface cracks in many cases do not extend beyond the surface film, yet by the authors' system of rating it might be assumed that such walls had lost 40 to 50 per cent of their design strength. It seems, therefore, that the entire scale of ratings could be revised and more liberal values for percentage of design strength be assigned to all but perhaps the two lowest ratings (H and I).

The use of single wall specimens for the exposure tests and of relatively few companion cylinders for strength tests is, no doubt, partly responsible for the lack of consistent relationships between strength and durability and between strength and water-cement ratio. In the case of walls 2 to 6, inclusive, the water ratio is indicated to be the same while the compressive strength at 28 days varies from 2759 to 4138 lb. per sq. in. and the authors' durability rating from 50 to 80. Again, specimens 26, 27 and 28 have a spread in water-cement ratio of 0.74 to 0.86 by volume but have almost identical strengths, and thus far have shown practically the same resistance to weathering. It is not stated in the paper in what order the walls were made or the methods employed in casting, finishing, and curing them. Consequently it is impossible to judge whether or not some of the inconsistencies observed may have been due to differences in workmanship and inaccuracy of control. The use of fixed proportions over a considerable range in water-cement ratio is known to result in unbalanced mixtures in some part of this range. This would be especially true of the $1:2\frac{1}{2}:5$ mixture with the higher water-cement ratios. Any difference in procedure in disposing of the excess water which would inevitably rise to the top of a 3-ft. form could easily overshadow the effects of other factors on the durability of the walls. It is significant that with few exceptions the L's or offset lower portions of the walls are in excellent condition. Moreover, temperature differences during placing and early hardening may have influenced the behavior of the walls, since, according to the temperature chart for 1934, shown by the authors, the temperature during the month of September (the month in which the specimens were made) is subject to a fluctuation from a maximum of 90 F. to a minimum of 30 F., with temperature ranges as great as 50 deg. occurring in a single 24-hr. period.

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[1] Manager, Research Laboratory, Portland Cement Assn., Chicago, Ill.

The chemical analyses and calculated compound compositions of the cements show so little difference that the writer believes that this is not a factor of major importance in these tests. The quality of the sand appears to have had a more important effect since sand C shows relatively poor results with cement A, whereas with sand D, approximately the same results were obtained with both cements.

Serious disintegration appears to have taken place only in the case of certain mixtures in the cement A - sand C combination, which according to the authors is the poorest combination of the four tested. For the remaining combinations, the difference in durability thus far apparent between the different specimens within each group is so small that practically all in each group can be said to be on a par. It seems reasonable to expect that when the exposure is continued for a longer period, the relative performance of specimens of different water-cement ratios will parallel that of the cement A - sand C combination.

MR. F. N. SPELLER.[1]—It would be interesting to know whether the disintegration of this concrete is from contact with the soil or atmosphere.

MR. H. S. MATTIMORE.[2]—One is buried and the other is in the air.

MR. SPELLER.—It is not disintegrated in the soil?

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[1] Director, Department of Metallurgy and Research, National Tube Co., Pittsburgh, Pa.
[2] Engineer of Tests, Pennsylvania State Highway Dept., Harrisburg, Pa.

MR. MATTIMORE.—No, it is not.

MR. SPELLER.—Freezing and thawing may be the principal factor. Mr. Mattimore's experiments should be quite valuable but more data and study may be necessary to arrive at the correct explanation of the failure of concrete under these particular conditions. Pennsylvania soils in that district are as a rule only mildly corrosive; at least, they have relatively little action on pipe lines compared with the soils farther west.

I notice the pH value is given as indicating the acidity. The National Bureau of Standards' soil corrosion investigation started off by putting a good deal of stress on pH value and wound up by stressing total acidity.

We have made water solubility tests of cement, the iron content being one of the principal variables; we find that the cements with the higher iron contents are less soluble in water.

MR. R. B. GAGE.[1]—New Jersey has been constructing concrete pavements since 1917. Some of them have been resurfaced, and others look rather sick, while again, others have a healthy, durable appearance. We have a fairly good record of the materials used in the majority of these pavements, as well as the equipment and methods of construction followed.

In order to determine the causes that produced the difference in the behavior of these pavements, a series of concrete cores were drilled from different pavements. The pavements selected were characteristic of concrete constructed with different materials, different methods of construction and types of subgrade, and subjected to different kinds of travel. The cores thus secured were brought to the laboratory and carefully examined, and their strength and density determined. The cores represented concrete from one year to ten years of age, and most of the cements and aggregates which have been used during this period.

In general, the results show that the concrete contained in pavements built with certain cements, sands and coarse aggregates has a considerably higher compressive strength today than those built with other cements, sands and aggregates, even when the construction conditions are similar. However, in other cases it was discovered that there is a marked difference in the condition of pavements that were constructed with the same cement, sand and aggregate, due, according to our records, to the low initial strength of the concrete, caused by the use of excess mixing water, abbreviated time of mixing, or combination of these two conditions.

The concrete in those pavements where excess mixing water was used has been found to be deficient in both strength and density, and since such concrete is not properly protected from attack, such porous concrete could not be expected to give satisfactory service. There is practically no hydrous silicate produced by nature that will resist decomposition when exposed to atmospheric agencies, and it is hardly to be expected that man is able to produce a hydrated silicate that is superior.

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[1] Chemical Engineer, New Jersey State Highway Dept., Trenton, N. J.

Portland cement is a hydrated silicate and the more and easier it can be attacked by surface waters, the quicker it is decomposed. Rain waters or snow waters will soon develop every one of the defects we have seen here today, and to prevent this decomposition is one of the principal reasons we are so particular in specifying a low density in our concrete pipe. In this particular case, we have thin sections of concrete and it is naturally to be expected that waters will soon tear such concrete to pieces unless it is properly protected.

There appear to be no data showing the relative rapidity with which rain or snow waters will decompose concrete. We know it is much in excess of ordinary ground water, which generally contains a certain amount of minerals. Naturally, freezing accelerates the rate of decomposition; however, in the southern States concrete pavements disintegrate and go to pieces where there is no frost action and such failures can only be caused by the decomposition that takes place in the cement itself by the action of surface waters.

The speaker feels that we are perfectly safe in assuming that unless concrete has sufficient density to protect itself from attack by atmospheric agencies, it will seldom give the service desired, but fortunately when the desired density is secured, such concrete usually has sufficient strength to permit it to function properly.

MR. MATTIMORE (*author's closure by letter*).—Mr. Anderegg mentions the possibility of sulfate action. In this regard specimens of both cements were subjected to sodium sulfate tests (Merriman test) and did not show any detrimental effect until the 6-months' period, at which time both specimens showed a slight curvature.

Mr. Scholer discusses the influence of poor aggregate. No doubt this is a factor and special attention is called to the effect of sand C, but it should be observed that this was influenced to some extent by the cement.

In regard to Mr. Gonnerman's comment on the rating, so far as we know this is the first attempt to rate extent of disintegration and failure. No doubt improvements will be made as students study these methods, but as to the surface checking due to rubbed mortar finish, this usually is attributed to the use of a steel trowel, while in the rubbed mortar finish on these walls a wooden trowel was used.

In regard to the differences between the cement as determined by chemical analysis and calculated compound composition, although the actual amount of C_4AF content is not abnormally high in either cement, the percentage difference of this compound in these two cements is quite high, and the main reason for calling attention to this compound is due to the tendency to increase such compound in order to reduce the C_3A content. Attention was called to the amount of alkalies in both cements from the standpoint that very little attention had been paid to these elements.

TESTS OF MORTARS FOR REINFORCED BRICK MASONRY

By M. O. Withey[1] and K. F. Wendt[1]

Synopsis

Tensile and compressive strengths, linear change measurements, absorption, freezing and thawing, and autoclave tests, also workability ratings, are reported. Most of the tests were made on 1:3 and 1:4 portland-cement mortars rendered more plastic by additions of lime or pulverized clay. The influence of five curing conditions, five lime hydrates, three varieties of clay, and differences in proportion of admixture are included. Approximately 82 mixes and 4000 test specimens are represented in the reported data. The large linear changes of the dolomitic lime-cement mortars under continuously wet curing and of the clay-cement mortars in dry air are shown. The data indicate the suitability of 1:4 mixes with proper addition of lime or clay for moderate exposure, but for high strength or severe exposure 1:3 or 1:2½ mortars with clay or lime additions appear preferable.

Introduction

The good strengths obtained from tests on reinforced brick masonry beams[2] and columns[3] aroused our interest in this type of construction. Since the strength and integrity of the bond between the mortar and the brick are prime essentials in these structural parts, it has seemed desirable to ascertain what mortars are satisfactory for making such members with various brick. To this end, tests have been and are still being made on the physical and mechanical properties of the mortars and brick and on tensile and shear bond strengths of assemblages of certain mortars and bricks in the Materials Testing Laboratory at the University of Wisconsin.

Herein are reported data on strength, linear change, absorption, workability, and durability, all very important properties for masonry mortars. These tests were made in the winter of 1933–1934, with the assistance of C.W.A. funds and a grant from the Wisconsin Alumni Research Foundation.

In the fall of 1933 the program initiated called for tension, compression, and expansion tests at 1, 3, 7, and 28 days. Near the end of this program, additional specimens were made to be tested at 3 months, 1, 2, and 5 yr. Owing to the stopping of the work earlier than had been anticipated, only about half of the specimens for the longer time program were made. Most of the mortars were made with one portland cement. Five lime hydrates and three powdered clays were used as plasticizers. A few tests are reported to show the results obtained with a masonry cement and a small number of tests on the effects of calcium chloride are included. Determinations of the effects of freezing and thawing and of autoclave tests on certain of the expansion specimens were also made. About 82 mortar mixes, 1550 tests of briquets, 1550 tests of 2-in. cubes, 800 expansion specimens, 130 absorption specimens, 114 freezing-and-thawing tests, and 100 autoclave tests are represented in this report.

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[1] Professor of Mechanics, and Instructor in Mechanics, respectively, University of Wisconsin, Madison, Wis.

[2] M. O. Withey, "Tests on Brick Masonry Beams," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 651 (1933).

[3] M. O. Withey, "Tests on Reinforced Brick Masonry Columns," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 387 (1934).

Credit is due Mr. C. F. Ewald for his careful supervision of tests and records; to Mr. L. L. Krasin, who made all specimens and assisted in the preparation of this report; and to Messrs. L. F. Marbes, E. F. Gahnz, and M. L. Burgeson for their assistance. Acknowledgment is also made to those manufacturers who donated products for these tests.

MATERIALS

Cements:

The tensile strength of 1:3 standard sand mortar briquets of the portland cement C averaged 395 and 470 lb. per sq. in. at 7 and 28 days, respectively. The cement passed soundness, fineness, and time of set requirements. Its magnesia content was 4.6 per cent.

In a series of tests on mortars containing calcium chloride admixtures a high early strength portland cement C_1 was also used. Its tensile strength in 1:3 standard sand mortar was 355 and 485 lb. per sq. in. at 3 and 7 days, respectively.

After preliminary tests on several masonry cements, the strongest, masonry cement D, which was sound and ground so that 99 per cent passed a No. 200 sieve, was included in the program of tests.

Limes, Clays, and Other Admixtures:

The results of tests on the five varieties of hydrated lime used follow:

Type	Designation	Soundness		Coarser Than No. 28 Sieve, per cent	Coarser Than No. 200 Sieve, per cent
		Boiling	Autoclave		
Dolomitic	L	fail	fail	1.1	9.3
High calcium	L_1	OK	OK	1.9	3.8
Dolomitic, special	L_2	...	...	4.7	31.8
Dolomitic, finishing	L_3	fail	OK	0.9	3.2
Dolomitic, low water ratio	L_4	OK	fail	5.3	28.7

Lime L_2 was hydrated in the Mining Laboratory by a special steaming process. Other varieties were commercial products. An asterisk (*) appended to a lime designation indicates the hydrate was soaked for 1 day prior to mixing. Double asterisks (**) indicate soaking for 7 days. Dry

TABLE I.—CLASSIFICATION AND PROPERTIES OF MORTARS.

C = Portland cement. S = screened Janesville sand (fineness modulus = 2.0).

Mix by Weight	Water-Cement Ratio, $\frac{w}{c}$, by Weight	Average Flow, per cent	Remarks
$1C:2S$	0.51	95	
$1C:3S$	0.65	80	
$1C:4S$	0.83	70	
$1D:2S$	0.50	75	D—A commercial masonry cement.
$1D:3S$	0.59	75	
$1L:3C:12S$	0.92	70	L—A hydrated dolomitic Wis. lime used dry.
$1L:2C:6S$	0.93	85	
$1L:5C:20S$	0.89	70	
$1L:3C:7\frac{1}{2}S$	0.75	80	
$1L:4C:16S$	0.90	75	
$1L:9C:36S$	0.89	75	
$1L^{*}:3C:12S$	0.91	75	L^{*}—Lime L soaked for 24 hours before mixing.
$1L^{*}:3C:7\frac{1}{2}S$	0.69	75	
$1L^{*}:4C:16S$	0.89	75	
$1L^{*}:5C:20S$	0.88	75	
$1L^{*}:9C:36S$	0.87	75	
$1L^{**}:3C:12S$	0.90	65	L^{**}—Lime L soaked 7 days before mix.
$1L_1:3C:12S$	0.92	75	L_1—A hydrated high-calcium lime used dry.
$1L_1:3C:7\frac{1}{2}S$	0.73	70	
$1L_1:4C:16S$	0.93	65	
$1L_1^{*}:3C:12S$	0.90	75	L_1^{*}—Lime L_1 soaked for 24 hours before mixing.
$1L_1^{*}:3C:7\frac{1}{2}S$	0.69	70	
$1L_1^{*}:4C:16S$	0.90	80	
$1L_1^{**}:3C:12S$	0.90	65	L_1^{**}—Lime L_1 soaked 7 days before mix.
$1L_2:3C:12S$	1.01	60	L_2—A dolomitic Wis. lime experimentally hydrated.
$1L_2:3C:7\frac{1}{2}S$	0.85	60	
$1L_2^{*}:3C:12S$	0.94	65	L_2^{*}—Lime L_2 soaked for 24 hrs. before mixing.
$1L_2^{*}:3C:7\frac{1}{2}S$	0.78	75	
$1L_3:3C:12S$	0.95	65	L_3—A hydrated Wis. dolomitic finishing lime used dry.
$1L_3:3C:7\frac{1}{2}S$	0.76	70	
$1L_3:4C:16S$	0.92	60	

Mix by Weight	Water-Cement Ratio, $\frac{w}{c}$, by Weight	Average Flow, per cent	Remarks
$1L_3^{*}:3C:12S$	0.96	65	L_3^{*}—Lime L_3 soaked for 24 hrs. before mixing.
$1L_3^{*}:3C:7\frac{1}{2}S$	0.76	70	
$1L_3^{*}:4C:16S$	0.88	60	
$1L_4:3C:12S$	0.88	65	L_4—A special low water ratio dolomitic hydrated lime.
$1L_4:3C:7\frac{1}{2}S$	0.64	65	
$1L_4:4C:16S$	0.88	60	
$1L_4^{*}:3C:12S$	0.85	60	L_4^{*}—Lime L_4 soaked for 24 hrs. before mixing.
$1L_4^{*}:3C:7\frac{1}{2}S$	0.62	65	
$1L_4^{*}:4C:16S$	0.86	65	
$1B:4C:12S$	0.67	80	B—A finely ground clay from the Waupaca, Wis., area.
$1B:6C:18S$	0.66	80	
$1B:10C:30S$	0.65	75	
$1B:4C:16S$	0.84	75	
$1B:5C:20S$	0.83	70	
$1B:9C:36S$	0.82	65	
$1B_1:4C:12S$	0.71	75	B_1—A finely ground clay from the Chicago, Ill., area.
$1B_1:10C:30S$	0.66	75	
$1B_1:4C:16S$	0.86	65	
$1B_1:9C:36S$	0.85	70	
$1B_1:5C:20S$	0.85	65	
$1B_1:6C:18S$	0.67	75	
$1B_2:4C:12S$	0.82	75	B_2—A commercial admixture consisting largely of clay.
$1B_2:10C:30S$	0.71	75	
$1B_2:4C:16S$	0.97	70	
$1C_1:3S$	0.63	70	C_1—Incor cement.
$1B:6C_1:18S$	0.66	70	B—See above.
$1L^{*}:3C_1:12S$	0.91	75	L^{*}—See above.
$1L^{*}:3C_1:7\frac{1}{2}S$	0.69	60	B_1—See above.
$1L^{*}:3C':12S$	0.91	75	C', C'_1, D'—Portland cement, Incor cement, and masonry cement tempered with water containing 1½ per cent calcium chloride by weight of cement.
$1L^{*}:3C':7\frac{1}{2}S$	0.69	60	
$1B:6C':18S$	0.66	80	
$1B_1:6C':18S$	0.67	75	
$1D':2S$	0.50	80	
$1B_1:6C_1:18S$	0.67	65	
$1C':3S$	0.65	80	
$1C'_1:3S$	0.63	80	
$1L^{*}:3C'_1:12S$	0.91	80	
$1L^{*}:3C'_1:7\frac{1}{2}S$	0.69	60	
$1B:6C'_1:18S$	0.66	80	
$1B_1:6C'_1:18S$	0.67	65	

hydraters L, L_1 and L_4 weighed, when rodded into measure, 33, 33, and 50 lb. per cu. ft., respectively.

Clay B from the Waupaca area was ground so that 99 per cent passed a No. 100 sieve and 65 per cent passed a No. 200 sieve. Clay B_1 from the Chicago area exhibited a fineness of 90 per cent passing the No. 100 sieve and 40 per cent passing a No. 200 sieve. Admixture B_2 was a commercial ground clay from Iowa—60 per cent passed a No. 48 sieve, 49 per cent passed a No. 100 sieve and 42 per cent passed a No. 200 sieve. Clays B, B_1 and B_2 weighed, rodded, 58, 54 and 69 lb. per cu. ft., respectively.

Sand:

Janesville mason's sand which contained material coarser than the Nos. 14, 28, 40, and 100 sieves by 5, 23, 76, and 98 per cent, respectively, was used in all tests. This sand weighed 107 lb. per cu. ft., rodded, and 101 lb. per cu. ft., loose.

MORTARS

Proportions:

All mortar proportions were measured by *weight*. Lime proportions are in terms of the *weight* of the dry hydrate as received. Proportions of admixture if designated by volume are much higher. For example: using the specific weights given and assuming cement at 94 lb. per cu. ft. the proportions equivalent to $1L_1{:}3C{:}12S$, by weight, are $1L{:}1.6C{:}5.6S$, by volume. The water-cement ratios were obtained by dividing the weight of water by the weight of cement with no allowance for the absorption of the sand (0.5 per cent). Table I shows data for the mixes upon which this report is based. The proportions were made richer in cement than is usual for ordinary masonry because such mixtures were deemed essential to obtain the bond strength required for reinforced brick masonry.

Mixing:

The dry materials in batches approximating 2000 g. were mixed until uniform in color, then tempered with sufficient water to produce the desired workability. The batch was then mixed with a 6-in. round-nosed trowel in a shallow granite iron pan for 3 min., allowed to stand 3 min., and again mixed for 1 min. This long mixing period was found essential in order to reduce irregularities in flow-table results, especially when lime admixtures were made.

Whenever soaked lime was used it was mixed in definite proportions with sufficient water to form a creamy paste and allowed to stand in a covered pail until wanted. The excess water was then decanted, the proportions of dry hydrate in the paste determined, and sufficient lime paste was then weighed in a mixing pan for a given batch. To this lime paste the mixture of dry sand and cement was added and the batch mixed until the lime paste was broken down into small units. The additional water required was then poured in and the batch mixed 4 min., allowed to stand 3 min., and again mixed for 1 min.

For certain tests 1.5 per cent of chemically pure granular calcium chloride in terms of the weight of the cement was dissolved in the mixing water.

Flow-Table Tests:

At least one flow per mix per day was run on a 10-in. mortar flow table which, during the major part of the tests, was given thirty $\frac{1}{8}$-in. drops in 30 sec. In the initial trials the flow ranged in most cases between 60 and 75. After determining the percentage of water from such trials, that percentage was used in subsequent batches of the given mix, regardless of later flow-table results. The reported flows are averages for the batches tested.

The workability ratings given the various mortars in Figs. 2, 5, and 6 were based largely upon the judgment of a single operator, with limited experience in building construction, who did all the mixing and flow-table testing. The chief factors upon which his classification was made were troweling qualities, stickiness, and homogeneity of the batch when it had a flow of 60 to 75. Some of the sticky mortars like the high-lime mixes and those with admixtures B_2 might have received a higher rating had they been tempered with less water and made with lower flows.

Making Specimens:

Briquets were molded in the standard manner. The 2-in. cube molds were half-filled, the mortar tamped 25 times with a bullet-nosed tamper $\frac{5}{8}$-in. in diameter, and 6 in. long, filled, and the tamping operation repeated. Top surfaces were then troweled smooth and level.

Prisms for determining the linear changes due to various curing conditions were made by puddling the mortar into 1 by 1 by 6-in. cold-rolled steel molds through a 6 by 1-in. face, inserting brass lugs into the center of each end, and then striking off the excess mortar with a trowel.

Curing Conditions:

All specimens were placed in the molds in a moist closet immediately after making. After 24 hr. the molds were removed and the specimens marked and subjected to one of the following curing conditions:

Curing Condition 1.—Specimens were cured in moist closet until tested.

Curing Condition 2.—After removal from molds specimens were cured on racks in the air of the laboratory; approximate humidity range 30 to 40 per cent, average 35 per cent.

Curing Condition 3.—One day in molds in moist closet, 6 days in laboratory air, 3 days moist, balance in air.

Curing Condition 4.—In moist closet for 28 days, then cycles consisting of 11 days in air and 3 days in moist closet until 6 months old, followed by cycles consisting of 3 days in moist closet, balance of each month in air.

Curing Condition 5.—One day in molds in moist closet, 6 days in laboratory air, 3 days moist, 18 days in air, then cycles consisting of 3 days in moist closet, balance of each month in air until 6 months, then 3 days in moist closet every 3 months, the balance in air.

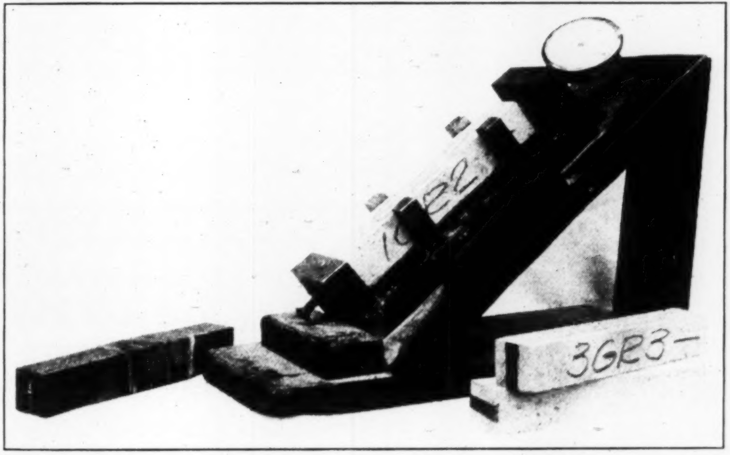

FIG. 1.—Apparatus for Measuring Linear Changes of 1 by 1 by 6-in. Prisms.

Testing Specimens:

Briquets were broken in an approved Olsen automatic shot briquet testing machine at standard speed.

Cubes were loaded on side surfaces at free speeds of head of 0.018 to 0.027 in. per min. Before testing, the bearing surfaces of the cubes were scraped level within 0.01 in.

All specimens were broken wet except in curing conditions 2 and 3.

Expansion and contraction readings were taken immediately on removal of the prism molds and at frequent intervals thereafter by means of the apparatus shown in Fig. 1. This device and the 1 by 1 by 6-in. steel gage bar were allowed to secure the temperature of the room in which the readings were taken. The dial was set to read zero on the gage bar and readings were then taken on the specimens. At 1 yr. the reported contractions were taken on air-dry specimens.

Autoclave tests to ascertain the expansion of mortar prisms were made in a long cylindrical pipe with internal dimensions $8\frac{3}{4}$ by 58 in. Steam at 150 lb. per sq. in. pressure was slowly admitted and the pressure maintained for 3 hr. This autoclave was also used in making the soundness tests on the limes, but the pressure was then 120 lb. per sq. in. and the time 2 hr.[1]

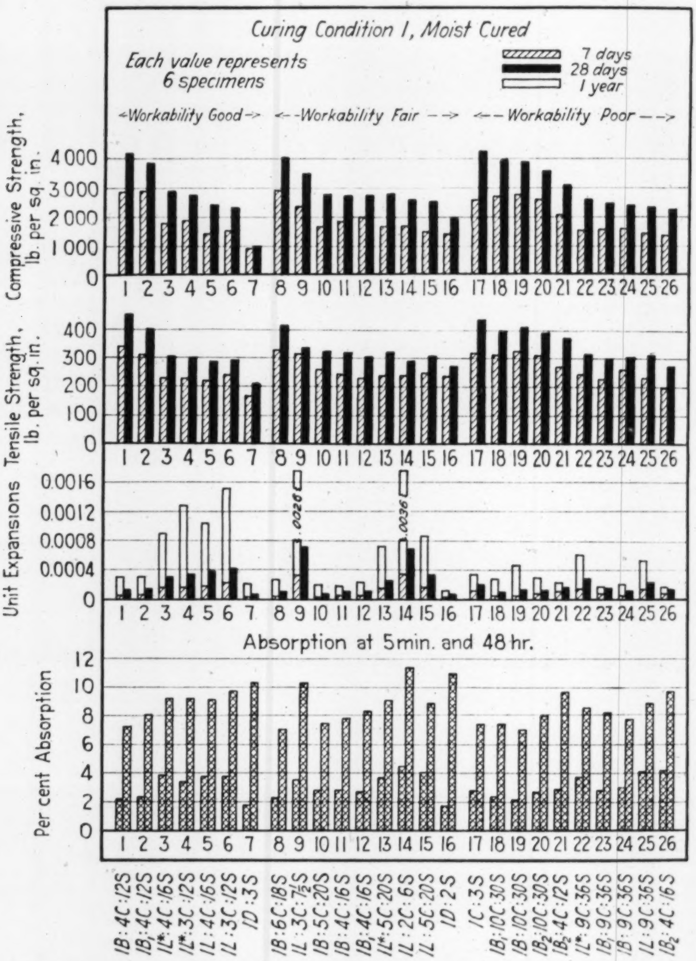

FIG. 2.—Strength-Age, Expansion-Age and Absorption-Time Relations for Curing Condition 1.

Freezing-and-thawing tests were conducted on certain of the expansion prisms after curing in various ways for 1 yr. In this test the prisms were immersed in water for 3 days, then placed in a tray in water $\frac{1}{2}$ in. deep and frozen. It is estimated from records on other prisms and cylinders that these specimens became frozen within 3 hr. and reached −7 F. within

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[1] See Tentative Methods of Physical Test for Limestone, Quicklime and Hydrated Lime (C 110 - 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 754 (1934); also 1934 Book of A.S.T.M. Tentative Standards, p. 310.

11 hr. after placing in the freezer. After 16 hr. in the freezer, specimens were removed and thawed by filling the tray with running water at 65 to 70 F. Expansion readings were taken after every ten cycles.

Absorption tests were made on the broken briquets which had stood in air for 1 to 5 months. The briquets were dried for 16 hr. at a temperature just above the boiling point of water and then weighed. Absorptions at 5 min. and 48 hr. are reported for curing condition 1, only.

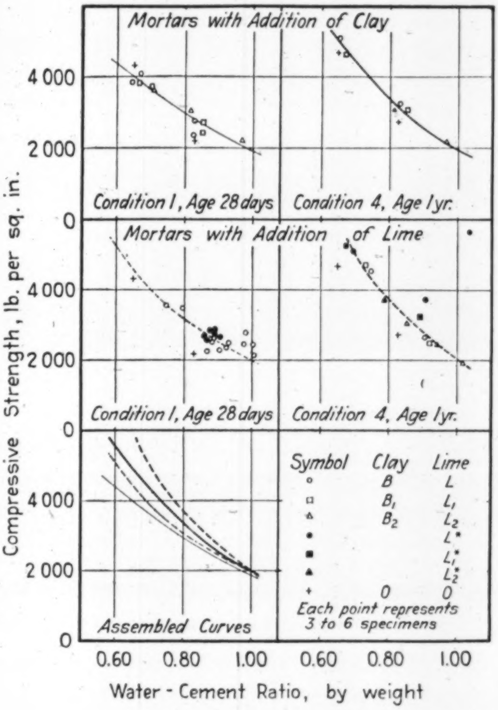

FIG. 3.—Compressive Strength Water-Cement Ratio Relationships for Mortars Under Curing Conditions 1 and 4.

TEST RESULTS

Effect of Curing Conditions:

The results of 7 and 28-day tension and compression tests; expansion determinations at 7 days, 28 days and at 1 yr.; and absorption tests after immersing 5 min. and 48 hr. for specimens of 26 mortar mixes, are given for curing condition 1 in Fig. 2.

For structures in which reinforced brick masonry might be used, curing conditions 1 and 2 represent extreme degrees of wetness and dryness whereas the other curing conditions approximate more nearly the usual conditions in building construction. Since strength standards for mortars and concretes of hydraulic cements have been based upon moist curing at

70 F., curing condition 1 serves as a basis both for ascertaining the effect of moisture and also for making strength comparisons. Assuming for reinforced brick masonry that a mortar with a flow of 60 to 75 should possess a tensile strength of 250 lb. per sq. in. and in cube form a compressive strength of 2200 lb. per sq. in. when tested after moist curing for 28 days,

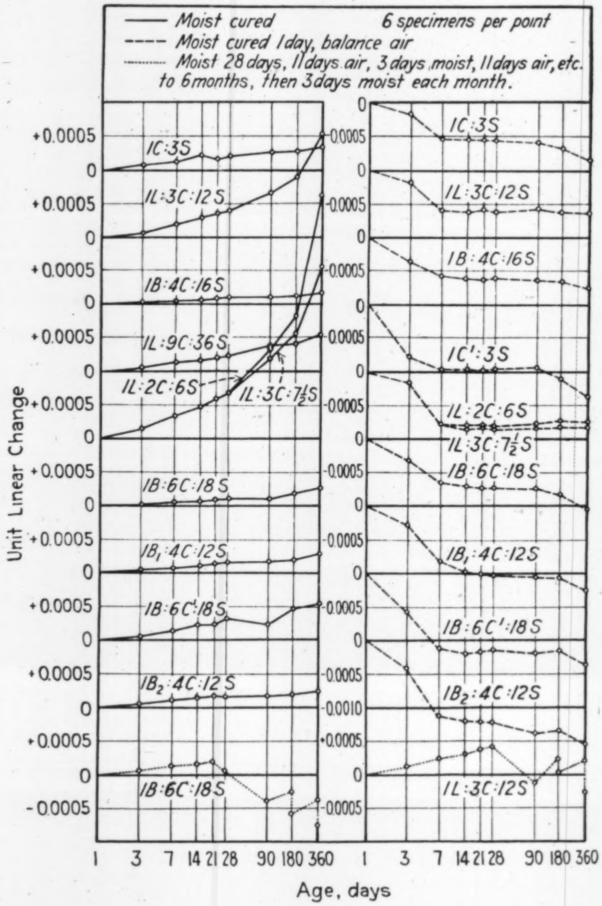

FIG. 4.—Effect of Age of Linear Change in 1 by 1 by 6-in. Mortar Prisms Variously Cured.

it will be noted that practically all of the mortars tested carrying 1 part of portland cement to 4 parts of sand, by weight (1:4) plus a clay or lime admixture for plasticizing were sufficiently strong for the purpose. Where higher strength is demanded the 1:3 portland cement mortars with like flows and suitably plasticized gave 28-day tensile strengths in excess of 350 lb. per sq. in. and compressive strengths above 3500 lb. per sq. in.

Comparing the data for the 1L:2C:6S dolomitic lime cement mortar with that for 1C:3S mortar it appears that the lower strength of the former mix was due to the addition of too much lime. Figure 3 shows the strength-water-cement relationships for 28-day strengths under curing condition 1 and for 1-yr. strengths with curing condition 4. The assembled curves indicate that with a given water-cement ratio the lime-cement mortars were slightly stronger than the clay-cement mixtures.

Under continuously moist curing the cement mortars tempered with clay showed small expansions, in most cases less than that of the 1C:3S portland cement mortar. On the other hand, mortars tempered with high percentages of dolomitic lime under continuous moist curing exhibited excessive expansion, which was probably due to the high magnesia content of these mortars. A better concept of the effects of age and curing condition on the linear change of typical mortars may be had from Fig. 4. The data from the absorption tests indicate that the range in absorption after 48 hr. immersion was from 7 to 11 per cent, that the high-strength mortars containing clay absorbed the least, and that those containing high percentages of lime and the masonry cement mortars absorbed the most. Absorptions for curing conditions 2 and 3 were essentially the same as for condition 1.

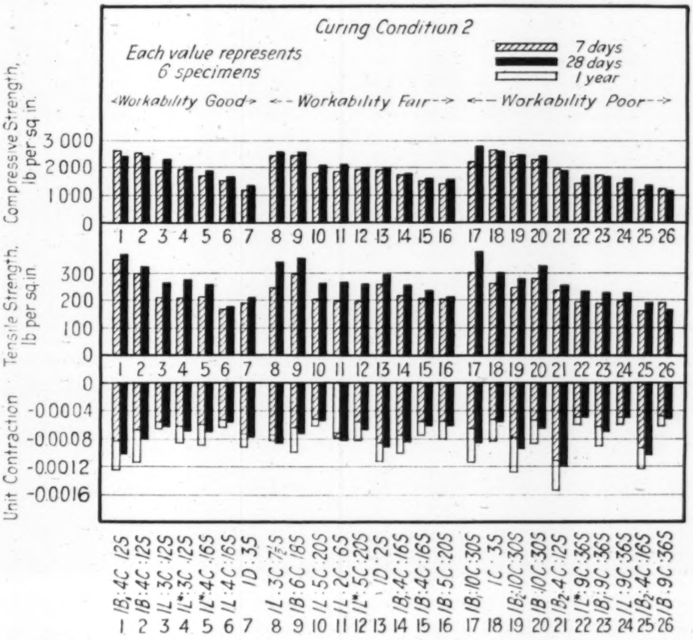

FIG. 5.—Strength-Age and Contraction-Age Relations for Curing Condition 2.

Under condition 2, air curing after 1 day in molds, the 7-day mortar strengths shown in Fig. 5 approximated those obtained in moist curing at the same age but the diagram indicates there was little change in compressive strength between 7 and 28 days. Some of the mixes carrying high cement and high clay contents showed slight retrogressions in compressive strength between 7 and 28 days. The tensile strength of the tested mixes showed increases between 7 and 28 days but usually of small magnitude.

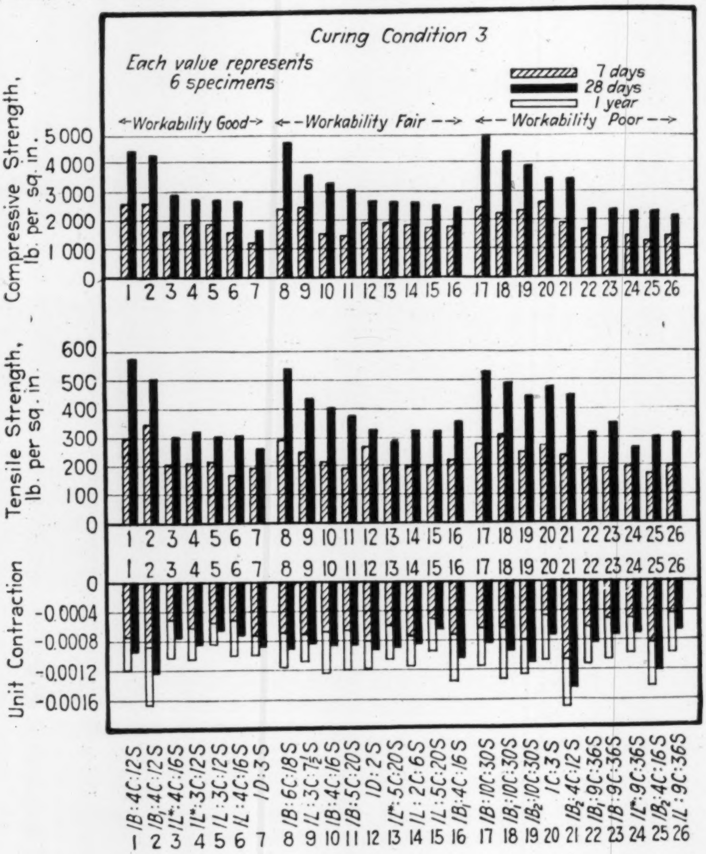

FIG. 6.—Strength-Age and Contraction-Age Relations for Curing Condition 3.

Mortars of 1:3 proportions containing high clay contents showed rather high contractions under this condition whereas the mortars tempered with hydrated lime exhibited less contraction.

Figure 6 shows that under curing condition 3, partly moist, the mortars containing clay admixtures exhibited higher 28-day strengths than in curing condition 1. Mixes containing lime were of about the same strength, while the 1C:3S portland cement mix had considerably less compressive strength than in curing condition 1. Prisms of nearly all mortars exposed to condition 3 suffered greater contractions than companion specimens exposed to air curing in condition 2.

Additional tests under condition 3 were also made on cement mortars containing high-calcium hydrated lime L_1, dolomitic finishing hydrated lime L_3 and a low water ratio dolomitic hydrated lime L_4. The strength and contraction data for mixes made with soaked and with dry hydrates are shown in Fig. 7. Considering the data in both Figs. 6 and 7 it will be observed that the mortars made with dolomitic hydrate L_4 in 1:3:7½ proportions had strengths equal to the best of the clay-cement mixes and considerably better than any of the other lime-cement mixes. In like mixes of lower cement content made with hydrates L, L_3 and L_4 the strengths were more nearly equal. Mortars made with the dolomitic limes were stronger than those of like proportions made with high-calcium hydrate L_1. In nearly all cases, soaking the hydrate one day prior to mixing produced stronger mortar. With a given cement-sand ratio, the mortars having higher lime contents contracted somewhat more than those with lower lime contents.

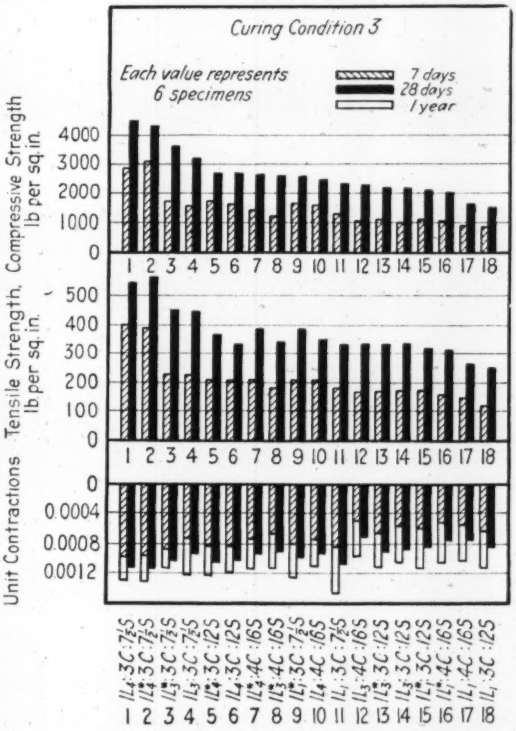

FIG. 7.—Strength-Age and Contraction-Age Relations for Certain Lime Mortars.

Of the mixes subjected to curing condition 4 and tested at one year, see Fig. 8, the $1L_1:3C:7\frac{1}{2}S$ and $1L:3C:7\frac{1}{2}S$ high-calcium and dolomitic lime-cement mortars, also the 1B:6C:18S clay-cement mortar, exhibited very good compressive strengths. The lime-cement mortars made with

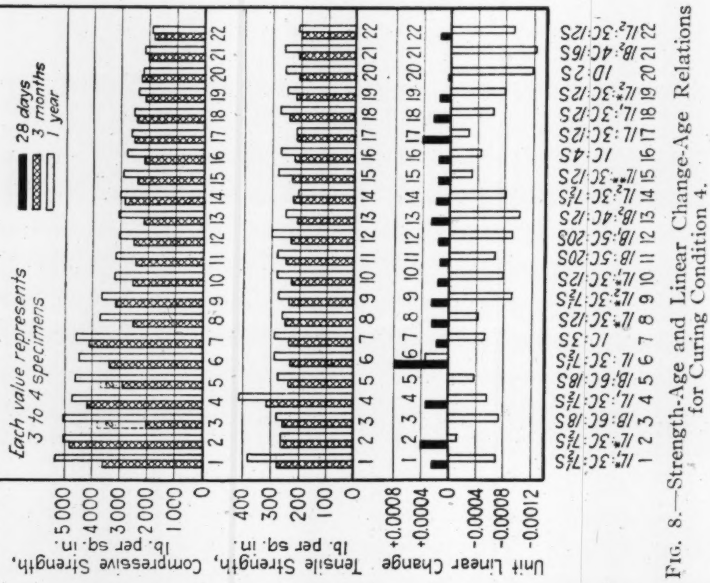

FIG. 8.—Strength-Age and Linear Change-Age Relations for Curing Condition 4.

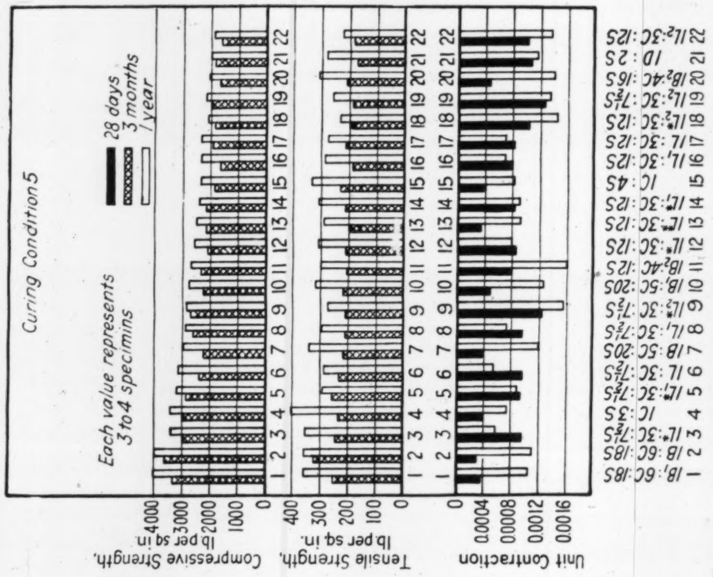

FIG. 9.—Strength-Age and Contraction-Age Relations for Mortars in Curing Condition 5.

soaked high-calcium or soaked dolomitic lime were somewhat superior in compressive strength and somewhat less strong in tension than corresponding mortars made with unsoaked lime. Tensile strengths at 1 yr. under this curing condition were lower than under condition 1 or 3 at 28 days. Contractions at 1 yr. in the lime-cement mortar prisms were less than in the clay-cement mortar prisms and both contracted less than in curing conditions 2 or 3.

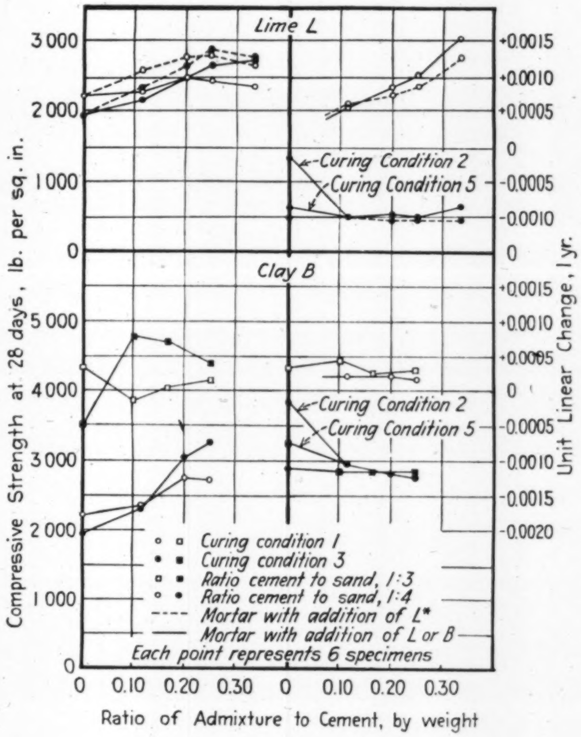

FIG. 10.—The Effect of the Proportion of Lime or Clay Admixtures on Strength and Linear Change of Cement Mortars.

When subjected to curing condition 5, with less frequent moist periods than in condition 4, Fig. 9 shows that the compressive strengths of the mortars were in general lower than in condition 4 and the tensile strengths slightly higher. The contraction of the clay-cement mortars under condition 5 was about the same as in curing condition 3 after 1 year, and the contraction of the lime-cement and other mortars was less than in condition 3. In general, contractions at one year were considerably greater under curing condition 5 than under condition 4.

Influence of Proportion of Admixture:

The influence of the proportion of the admixture of lime or clay on the compressive strength and linear change of cement mortars is shown in Fig. 10. These data also clearly show that mortars made with pre-soaked hydrate have somewhat greater compressive strengths than those made with unsoaked hydrate.

TABLE II.—EFFECTS OF 40 CYCLES OF FREEZING AND THAWING ON VARIOUS MORTARS CURED FOR ONE YEAR.

Curing condition 1—all moist.
Curing condition 2—one day moist, balance air.
Curing condition 3—1 day moist, 6 day air, 3 day moist, balance air.
††††† 5 to 20 per cent disintegrated.
§§§§§ More than 20 per cent disintegrated.
Each value represents two specimens.
Figures in table are last readable unit expansions.
Specimens were 1 by 1 by 6-in. prisms.

Mix by Weight	Curing Condition	10 cycles	20 cycles	30 cycles	40 cycles	Mix by Weight	Curing Condition	10 cycles	20 cycles	30 cycles	40 cycles
1B:4C:12S...	1				0.0017	1L:3C:$7\frac{1}{2}$S..	1	0.0030	††††††††	§§§§§§§§	§§§§§§§§
1B:6C:18S...	1				0.0012	1L:4C:16S...	1	0.0026	††††††††	§§§§§§§§	§§§§§§§§
1B:10C:30S..	1				0.0016	1L:5C:20S...	1	0.0026	††††††††	§§§§§§§§	§§§§§§§§
$1B_1$:4C:12S...	1			0.0023	§§§§§§§§	1L:9C:36S...	1	0.0018	††††††††	§§§§§§§§	§§§§§§§§
$1B_1$:10C:30S..	1			0.0014	§§§§§§§§	$1L^{\bullet}$:4C:16S..	1	0.0015		††††††††	§§§§§§§§
$1B_2$:4C:12S...	1		0.0048	§§§§§§§§	§§§§§§§§	$1L^{\bullet}$:5C:20S..	1		0.0022	††††††††	§§§§§§§§
$1B_2$:10C:30S..	1		0.0030	††††††††	§§§§§§§§	$1L^{\bullet}$:9C:36S..	1	0.0009	††††††††	††††††††	§§§§§§§§
1B:4C:16S...	1			0.0035	§§§§§§§§	1L:3C:$7\frac{1}{2}$S..	2	0.0008	§§§§§§§§	§§§§§§§§	§§§§§§§§
1B:5C:20S...	1		0.0021	††††††††	§§§§§§§§	1L:3C:12S...	2	0.0005	††††††††	§§§§§§§§	§§§§§§§§
1B:9C:36S...	1		0.0018	††††††††	§§§§§§§§	1L:4C:16S...	2		0.0003	††††††††	§§§§§§§§
$1B_1$:4C:16S...	1		0.0024	††††††††	§§§§§§§§	1L:5C:20S...	2		0.0004	††††††††	§§§§§§§§
$1B_1$:9C:36S...	1		0.0019	§§§§§§§§	§§§§§§§§	1L:9C:36S...	2				0.0003
$1B_2$:4C:16S...	1	0.0028	§§§§§§§§	§§§§§§§§	§§§§§§§§	$1L^{\bullet}$:3C:12S..	2				0.0003
1B:4C:12S...	2				0.0002	$1L^{\bullet}$:4C:16S..	2				0.0003
1B:6C:18S...	2				0.0002	$1L^{\bullet}$:5C:20S..	2		0.0006	††††††††	§§§§§§§§
1B:10C:30S..	2				0.0002	$1L^{\bullet}$:9C:36S..	2	0.0007	††††††††	§§§§§§§§	§§§§§§§§
$1B_1$:4C:12S...	2				0.0001	$1L_1$:3C:$7\frac{1}{2}$S..	3		0.0018	§§§§§§§§	§§§§§§§§
$1B_1$:10C:30S..	2				0.0002	$1L_4$:3C:$7\frac{1}{2}$S..	3				0.0004
$1B_2$:4C:12S...	2		0.0003	††††††††	§§§§§§§§	$1L_1$:3C:12S..	3	0.0004	††††††††	§§§§§§§§	§§§§§§§§
$1B_2$:10C:30S..	2			0.0003	††††††††	$1L_4$:3C:12S..	3				0.0005
1B:4C:16S...	2				0.0002	$1L_1$:4C:16S..	3	0.0003	††††††††	§§§§§§§§	§§§§§§§§
1B:5C:20S...	2				0.0002	$1L_4$:4C:16S..	3				0.0006
1B:9C:36S...	2				0.0001	$1L_1^{\bullet}$:3C:$7\frac{1}{2}$S.	3	0.0004	††††††††	§§§§§§§§	§§§§§§§§
$1B_1$:4C:16S...	2				0.0003	$1L_4^{\bullet}$:3C:$7\frac{1}{2}$S.	3				0.0003
$1B_1$:9C:36S...	2				0.0009	$1L_1^{\bullet}$:3C:12S.	3		0.0005	§§§§§§§§	§§§§§§§§
$1B_2$:4C:16S...	2		0.0005	§§§§§§§§	§§§§§§§§	$1L_4^{\bullet}$:3C:12S.	3				0.0004
1D:2S........	1				0.0006	$1L_1^{\bullet}$:4C:16S.	3	0.0003	††††††††	§§§§§§§§	§§§§§§§§
1D:2S........	2				0.0001	$1L_4^{\bullet}$:4C:16S.	3				0.0004
1C:3S........	2				0.0001						

Although the effect of the lime content on the strength of lime-cement mortars was not great, these data indicate an increase in compressive strength with additions of dolomitic hydrate up to 25 per cent of the weight of cement. Some data for mortars with additions of the high-calcium hydrate indicate that there was an increase in strength for additions of pre-soaked high calcium hydrate up to 33 per cent and a slight decrease for additions of dry high-calcium hydrate. The expansion at one year of the dolomitic lime-cement mortars under moist curing was nearly a linear function of the percentage of the hydrate reaching 0.0015 for the 1L:3C:12S mortar at 1 yr.

Figure 10 also shows that the compressive strength of 1:4 mortars was raised materially by additions of clay up to 25 per cent of the cement. For the 1:3 mixes clay additions to moist-cured mortars caused small reductions in strength, but under curing condition 3 the effect of such clay additions on strength was beneficial. The proportion of clay added to these cement mortars did not have much influence on the linear changes at 1 yr.

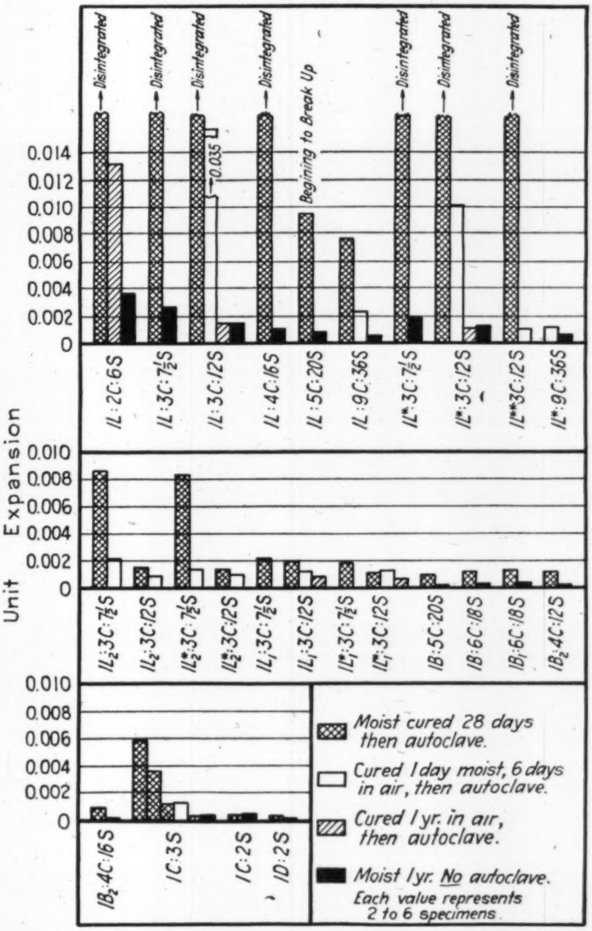

FIG. 11.—Effect of Autoclave Tests on Mortars Variously Cured.

Resistance to Freezing and Thawing:

Table II shows results of freezing and thawing tests made on 1 by 1 by 6-in. mortar prisms 1 yr. old. These data indicate that moist-cured mortar specimens had less resistance to freezing and thawing than similar air-cured specimens, that the 1:3 mixes with clay additions were superior to the 1:4 mixes with like additions, and that the clay-cement mortars withstood freezing and thawing better than the lime-cement mortars of equal cement content. Mortars containing clay B and those containing dolomitic hydrate **L_4** were the best of their respective types.

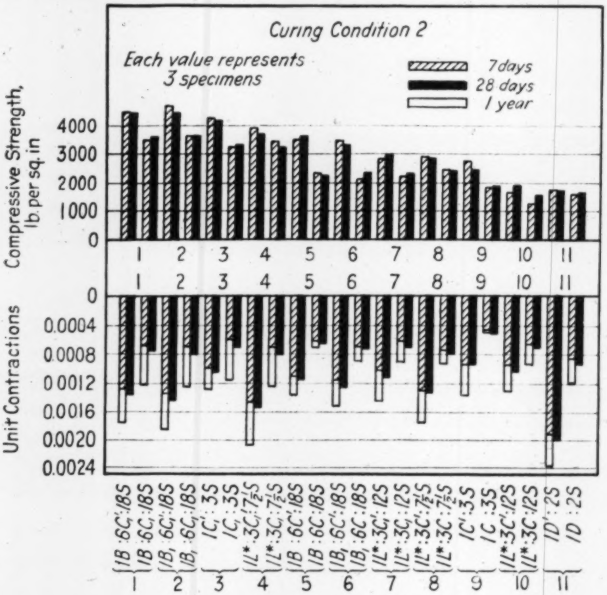

FIG. 12.—Strength-Age and Contraction-Age Relations for Mortars With and Without Calcium Chloride Admixtures.

Autoclave Tests on Mortars:

Autoclave tests were made to see whether there was any connection between the excessive expansions of the dolomitic lime-cement mortars under moist curing and their behavior in the autoclave, also to ascertain how various curing conditions prior to the autoclave test would affect the results of that test. Figure 11 shows expansion data obtained from tests on one hundred 1 by 1 by 6-in. prisms tested as indicated. With dolomitic hydrate L, moist-cured mortars having a hydrate-cement ratio of 11 per cent expanded about $\frac{3}{4}$ per cent in the autoclave and mortars with a ratio of 25 per cent or more disintegrated. Similar moist-cured mortars of high-calcium hydrate L_1 and portland cement expanded less than $\frac{1}{4}$ per cent in the autoclave. When mortars containing dolomitic hydrate L with hydrate-cement ratios of 33 per cent or less were air-cured 1 yr. and then tested in the autoclave they showed less than 0.2 per cent expansion. Also if the dolomitic hydrate L was soaked for a week prior to mixing and the specimens air-cured for six days after removal from molds, the expansion in the autoclave of mortars containing dolomitic hydrate-cement ratios of 33 per cent or less was only a normal amount. The expansion of mortars containing 11 per cent of dolomitic hydrate L by weight of cement was rendered negligible either by soaking the hydrate 1 day prior to use or by air curing for six days after removal from molds. The experimentally prepared hydrate L_2 gave lower expansions in the autoclave than commercial product L, but hydrate L_1 seemed to lower the strength of mixes in which it was included. The clay-cement mortars, portland-cement mortars and the masonry cement mortar all passed the autoclave test in satisfactory manner.

Effect of Calcium Chloride:

The results of compressive strength and contraction tests on 11 mortars with and without 1½ per cent of calcium chloride under curing condition 2 are shown in Fig. 12. The addition of this accelerator produced increases in 7-day strength of 15 to 35 per cent with the high early strength cement and of 20 to 50 per cent with the portland cement. Under this curing condition there was little change in strength between 7 and 28 days for mixes with or without the accelerator. The addition of the accelerator increased contractions at all ages from 50 to 100 per cent.

CONCLUSIONS

1. Mortars of 1 part portland cement to 4 parts sand, by weight, tempered with 25 to 33 per cent of hydrated lime or 20 to 25 per cent of very finely ground clay are sufficiently workable and strong for most reinforced brick masonry under moderate exposures. Where high strength is wanted or the exposure is severe, a 1:2½ mortar with 25 to 33 per cent of hydrate or a 1:3 mortar with 17 to 25 per cent of clay is tentatively recommended.

2. Soaking the hydrated lime prior to mixing in nearly all cases rendered the mortar stronger.

3. Mortars made with dolomitic hydrates were stronger than those made with the high-calcium hydrate.

4. Mortars of the low water ratio dolomitic hydrate were more resistant to freezing and thawing than the other lime-cement mortars tested.

5. Dolomitic lime-cement mortars containing over 11 per cent of hydrate by weight of cement expanded unduly when continuously wet or when moist cured and tested in an autoclave. Autoclave tests indicate that by pre-soaking the lime for a week and air curing the mortar for a week the expansion may be made normal.

6. Under dry curing conditions, lime-cement mortars with the higher lime contents contract slightly more than the portland cement mortars.

7. The clay-cement mortars were somewhat less workable, exhibited lower expansions under moist curing, but under air curing contracted somewhat more than the lime mortars.

8. The use of calcium chloride to increase the early strength of a mortar is attended by a marked increase in contraction under air curing.

9. The results indicate the desirability of ascertaining the properties of cement mortars containing mixtures of both lime and clay. The magnitudes of the linear changes of the lime-cement and clay-cement mortars in certain curing conditions necessitate determinations on the durability of the bond of these mortars to brick.

DISCUSSION

MR. J. W. MCBURNEY.[1]—I should like to ask Mr. Withey whether he has any information on the degree of hydration of the magnesia portion of the dolomitic hydrates which showed large volume expansions.

MR. M. O. WITHEY.[2]—I have not. I expect it was not complete by a considerable amount, but that is an assumption which is based upon other tests made in another laboratory.

MR. D. E. PARSONS.[3]—Many questions have been answered about the properties of the high strength plastic mortars that are apt to be valuable for use in reinforced brick masonry. At the same time questions that will be a challenge to research workers in this field have been raised. I have in mind particularly the large expansion that was found with some of the lime mortars and the large contractions exhibited by some of the mortars containing clay and calcium chloride. I hope Mr. Withey will continue his work and find the reasons for and the effects of these phenomena.

MR. J. W. GINDER.[4]—Those engaged in building, are interested in getting brick walls strong enough and also weatherproof. It seems strange that we should have so much trouble in getting weatherproof walls now when years ago we apparently did not have the same trouble.

To overcome this trouble some of us think more attention should be given to the mortars. Volumetric change and workability are important factors. The richer the mortar the more shrinkage, leaving cracks for water to enter.

Workability of the mortar for psychological as well as practical reasons is advisable. A mason does not like to use stiff mortar. It is more difficult to work and, therefore, he takes less interest in doing his work, feeling that it is an unnecessary hardship imposed upon him. The practical reason is that the more workable the mortar the more completely the joints will be filled.

The shrinkage of mortar at the joints and the lack of mortar in the joints is probably the greatest cause for masonry not being weatherproof. Therefore, our object should be to get a mortar with the minimum volumetric change and greatest workability possible that will give the strength necessary. After we have sufficient strength there is no need for more, and consideration should be given to other features.

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[4] Superintendent, Architectural Engineering, Engineering Section, Procurement Division, Public Works Branch, Treasury Dept., Washington, D. C.

MR. C. P. DERLETH.[1]—I should like to ask Mr. Withey whether any investigations have been conducted on cement mortars utilizing puzzolanic materials, having the capacity of combining with lime, as regards volume change and strengths?

MR. WITHEY.—We have not gone into that subject to any extent. We did make a few preliminary tests at the time this work was started.

MR. F. O. ANDEREGG[2] (*by letter*).—A valuable contribution has been made by Messrs. Withey and Wendt on the effect of lime and clay admixtures in masonry mortars. It is believed that somewhat similar relative results are to be expected with mortars spread out in comparatively thin layers on brick which remove moisture from the mortar, permitting additional compacting and giving lower water-cement ratios. The actual strength and durability of such mortars would doubtless be even better than the results given in this paper and the shrinkage would be considerably reduced. When a thin layer of one material is applied over another more massive material, provided the adhesion is satisfactory, the thin layer acquires to a considerable extent the properties of the base. The latter has a reinforcing action on the applied material and the thinner the coating, the greater the effect. This means that mortar in a thin joint approaches the brick in strength, durability and shrinkage properties. The desirability of much more work upon masonry mortars actually applied to brick surfaces cannot be too greatly emphasized.

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PRE-STRESSED CERAMIC MEMBERS

By F. O. Anderegg[1] and C. L. Dalzell[2]

Principle and Theory

Pre-stressing suggests itself for overcoming the tensile weakness inherent in unreinforced masonry. The principle of applying a force to various structures ranging from glass vessels to steel or concrete bridges, opposite to that received in normal loading has been utilized in the past to a limited extent, and will doubtless be more extensively used when the savings in materials, that may be obtained, are realized. When it is appreciated that ceramic units have been produced with compressive strengths[3] as high as 25,000 lb. per sq. in., while the tensile strength, when assembled into members, is of the order of 100 lb. per sq. in., it might seem reasonable to try to increase the latter at the expense of part of the tremendous resistance to crushing of such units, by some suitable means.

This can be accomplished by placing the whole member under compression, as by stretching steel rods running lengthwise and bearing on end plates. When the member is subject to bending in either direction, the result is a better balance between tensile and compressive strengths and ability to withstand forceful impact and heavy transverse loading. This principle has been applied to beams and to columns as described below.

Where a beam is to be loaded in one direction only, the pre-stress can be concentrated to a greater or less extent, depending upon the dimensions of the section, near the side receiving the tensile load. When the lower side of the beam is brought under compression by pre-stressing, a camber is produced in the beam and the neutral axis is shifted toward the top and may even pass outside the section. With a section having a vertical dimension of 10 in. or more, it may be necessary to place the upper part of the beam also under some initial compression to be sure that the tensile stress in the upper fiber, which accompanies the cambering, shall not exceed the bond strength of the mortar.

To match the high compressive strength obtainable commercially in ceramic units, a mortar so designed and applied as to approach the units in strength is desired and has been obtained.

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[2] Civil Engineer, Long Island City, N. Y.

[3] W. C. Rueckel and R. B. Keplinger, "The Effect of De-Airing, Dry-Press and Stiff-Mud Bodies for Clay Products Manufacture," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 480 (1934).

Considerable thought should be devoted to the choice of the reinforcing steel. Savings in weight justify the use of high yield point steel, especially where the cost is proportional to the yield point. It is believed, moreover, that a yield point greater than 100,000 lb. per sq. in., even though commanding some premium in price, will for certain purposes give the best balance of design, permitting the use of the materials to the best possible advantage to yield ceramic sections of lightest weight, a point of importance during erection. The greater the strength of the steel, the smaller its cross-section required, permitting smaller holes in the ceramic units and less ceramic material to surround the holes. This might seem to have a decided additional advantage in requiring less force to produce a given pre-stress in the lower fibers and to develop greater load-carrying capacity and, especially, stiffness. The pre-stress also increases the shear resistance of the member.

To utilize these principles to the best practical advantage probably means mass production under controlled conditions either at the tile plant or at some suitable center for distribution. Such members offer promise of fitting in well with factory-fabricated buildings, offering fire-proofed and weather-resistant members of light weight and reliable load-carrying capacity.

Historical:

Leading up to this development, Jackson,[1] Lee,[2] Brinkman,[3] Lund,[4] Henderson,[5] and Jagdmann,[6] have extended reinforcing rods beyond the beams ends and have tightened nuts to hold the units together, but without pre-stressing, except that Jagdmann also put extra tension on the rods to "produce an arching effect" on the member as soon as assembled. However, this method fails to develop the compressive strength of the mortar on the upper part of the beam where it will be needed later. In precast concrete, Dill[7] and Hewett[8] have proposed to coat the reinforcing rods with bitumen so as to permit adjustment during the pre-stressing, while Freyssinet[9] has made a notable contribution by stretching rods in the forms and then pouring concrete members.

MATERIALS USED

Ceramic Units:

In fabricating building members according to this method, the first consideration is the units themselves. These are designed for the particular application and are extruded, preferable with de-airing, using a fire clay or shale which has demonstrated ability to develop high compressive strength. The units are made hollow, as far as practicable, to reduce weight and to facilitate drying and burning. The dimension of the rod holes is determined by the size of the steel rods needed, sufficient space being left around the rods for complete grouting. The higher the yield point of the steel, the smaller the rod and the smaller the hole would be, thereby reducing the ceramic material surrounding the hole and limiting the amount of pre-stressing steel required. The units are placed in the hottest part of the kiln and are burned at the highest practical temperature. Other materials of high compressive strength are also available.

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[1] P. H. Jackson, "Improvement in Girders," U. S. Patent 126,396, May 7, 1872.

[2] T. A. Lee, "Fireproof Floor," U. S. Patent 461,029, October 13, 1891; "Floor and Roof," U. S. Patent 500,208, June 27, 1893.

[3] S. G. Brinkman, "Fireproof Ceiling and Floor," U. S. Patent 707,201, August 19, 1902.

[4] J. G. F. Lund, "Straight Vault," U. S. Patent 1,028,578, June 4, 1912.

[5] Albert Henderson, "Concrete Building Construction," U. S. Patent Re. 16,452, November 2, 1926.

[6] K. E. W. Jagdmann, "Building Construction," U. S. Patent 1,891,597, December 20, 1932.

[7] R. E. Dill, "Manufacture of Reinforced Concrete," U. S. Patent 1,684,663.

[8] W. S. Hewett, "Method and Means of Tying Concrete," U. S. Patent 1,818,254.

[9] E. Freyssinet, "New Methods in Concreting," *Science et industrie ed. materiaux de construction publics,* Vol. 1, p. 1 (1933).

Reinforcing Rods:

It is believed that best balance in the designing of these members is secured with steel rods of high yield point, for the reason mentioned in the preceding paragraph. An alloy steel such as the S.A.E. No. 3140 nickel-chromium steel after proper heat treatment, develops a yield point of 100,000 lb. per sq. in. or more, with a suitable tensile strength. This type of steel has given very satisfactory results in the experiments described below. An S.A.E. thread is recommended to facilitate tightening the nuts during the pre-stressing operation. For end plates $\frac{1}{4}$ or $\frac{3}{8}$ in. steel of a shape similar to the cross-section of the ceramic units might be used with holes punched for the rods.

Mortar:

The following mix has given excellent results, the mortar acquiring hardness similar to that of the ceramic units: 15 parts by weight of air-floated shale which has been demonstrated as producing no deleterious effect on the cement; 85 parts of portland cement, preferably of high early strength; and 250 parts of well-graded clean sand which has been sifted through a No. 12 or 14 sieve. The clay might be replaced with well-soaked, dolomitic lime putty, but it is believed that the fine shale gives better results, especially in view of the well-known flexibility imparted by lime to mortar. Part of the sand may be replaced with coarse, properly graded[1] portland cement clinker where extremely high strengths are desired. The mortar should stand 1 hr. before using and should be used up by the end of the second hour. Mechanical mixing has an advantage. The mortar is used in two consistencies, the one relatively stiff for the joints and the other much thinner for the grouting operation.

ASSEMBLY

With the rods upright, mortar is placed on the end plate and the first unit is let down over the rods and pounded against the mortar to give a joint about $\frac{1}{8}$ in. thick. The holes are then grouted solid with the aid, preferably, of vibration. Mortar is placed on the end of the first unit and a second is applied with tapping and vibration. During the erection of the beam, care is taken to keep it straight and guides are of great help in this regard.

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[1] F. O. Anderegg, "The Grading of Aggregates. II—Application of Mathematical Formulas to Mortars," *Industrial and Engineering Chemistry*, Vol. 23, p. 1058 (1931).

If the beam can be assembled on a platform properly counterbalanced, the erection is speeded up. When the last unit is in place, the second plate is bedded in mortar, the threads are cleaned and carefully lubricated, and the nuts are turned down so that the mortar will be under compression during the curing operation. The beam may then be lowered and the joints thoroughly tooled to compact them at the edges. The beams are cured under moist conditions for a week or ten days at room temperature, or for a shorter period at a higher temperature, to develop high strength in the portland cement.

PRE-STRESSING

After curing, the beams are held rigid by means of clamps and the rods are stretched by turning down the nuts on the ends to a predetermined value, which is roughly approximated by giving the nuts a definite number of turns, making the final adjustment with the aid of a micrometer or dial mounted on a suitably rigid frame. The rods may be stretched to between 50 and 70 per cent of their yield point to good advantage. The end plates and the nuts may then be covered with mortar to protect them against corrosion, or this may be done on the job, depending upon the particular application.

EXPERIMENTAL

The first experiments were made with well-burned, standard-size face brick, with holes bored by hand after extrusion. These were uniformly pre-stressed with the aid of two $\frac{5}{8}$-in. rods to 1500 lb. per sq. in. The beams were very elastic and developed much greater load-carrying capacity than had been anticipated, and either edge of this beam could receive loading.

The next beams were made from hollow tile extruded to give an inverted tee and were pre-stressed on the lower side only. The particular shape was designed for flooring joists to be used in combination with other materials. These beams failed only under heavy loads and the mortar bond withstood the shock of the failure in a remarkable manner. The mortar appeared to be as hard as the ceramic units themselves. For longer beams and more effective pre-stressing, calculated compressive stresses in the extreme fiber as high as 7870 lb. per sq. in. were obtained, but there is some doubt concerning the validity of the calculation.

Another set of hollow-tile units was manufactured for sills for factory-fabricated houses with a projection at the bottom for carrying one end of the floor slabs. These were assembled for test purposes with three $\frac{3}{4}$-in. high yield point steel rods, one being near the top. After pre-stressing, these beams were loaded at four points. Strain gages were placed on top and underneath the ceramic material and the deflections were read with Ames dials.

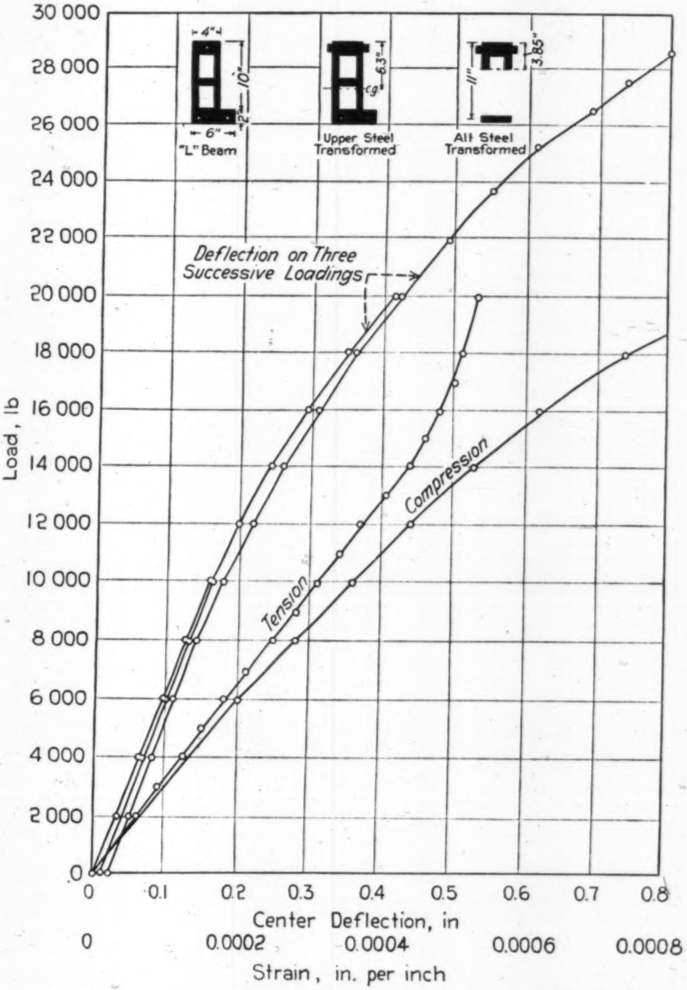

Fig. 1.—Test to Failure of Pre-Stressed L-Section.

The results of the observations with these gages are recorded in Fig. 1, as obtained for the beam $L-1$. To this beam a load of 5000, 10,000 and 20,000 lb. was successively applied with release each time. It was then broken at a load of 29,300 lb. Some idea of the change in length of the lower rods was obtained by means of an Ames dial which showed an elongation of a few thousandths of an inch, during the application of about the first half of the load. After this the stretching apparently accelerated reaching an additional value of about 0.1 in. at failure, which occurred in diagonal tension.

CALCULATIONS

The pre-stress in the steel is readily obtained from the measured elongation using the experimentally determined modulus of elasticity, 29,000,000 lb. per sq. in. The modulus of elasticity of the ceramic units was carefully determined for the lower loadings to be 6,120,000 lb. per sq. in., but diminishes, as may be seen from the compression curve in Fig. 1, thereby adding some uncertainty to the final stage of the analysis given below. The data are taken from the L-shape beam $L-1$.

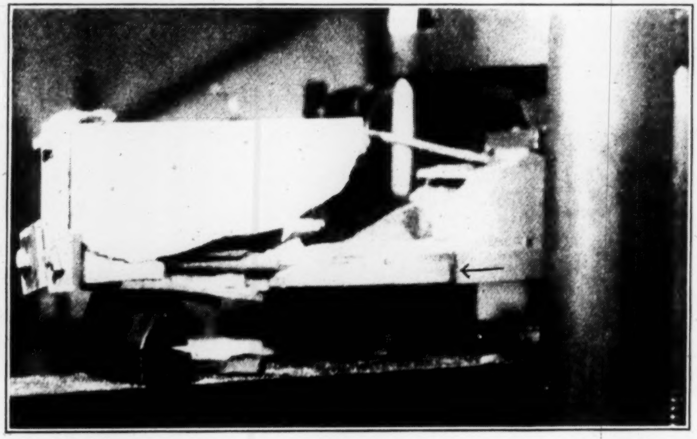

FIG. 2.—**Pre-Stressed Ceramic T Beam Failing in Shear Under Center Loading.** The arrow points to the mortar joints which at 7 days were practically as hard as the tile.

The following symbols are used:

f_u = ultimate unit stress in the upper ceramic fiber.
f'_u = pre-stress therein.
f''_u = unit stress in upper ceramic fiber when pre-stress in bottom fiber has been exactly neutralized.
f_b, f'_b and f''_b = the corresponding stresses in bottom ceramic fiber.
f_s, f'_s and f''_s = the corresponding stresses in the lower steel.
e = eccentricity of force P_s in lower steel.
$c_u = y$, $c'_u = y_1$, $c''_u = y_2$ = the distances of top fiber from neutral axes for the three stages, respectively.
c_b, c'_b, c''_b = distances from bottom fiber to neutral axes for the same three stages, respectively.

A_1 = for pre-stressing step, the area of the ceramic material plus upper steel transformed (lower steel is in tension) = 35.25 sq. in.

A_2 = for step during relief of pre-stress, the bottom steel rods as well as the upper rods and all the ceramic being now active = 39.45 sq. in.

A_3 = for final step, assume no tension in ceramic material below 6.5 in. from top of section = 23.09 sq. in.

I_1, I_2, and I_3 = moments of inertia for the three corresponding stages, pre-stress, relief of pre-stress, and final bending.

Step I. Pre-stressing Effect on Ceramic Material.—By the usual processes of summation, $y_1 = 6.2$ in. and $I_1 = 589$ in.^{4}

$$f'_u = \frac{-P_s}{A_1} + \frac{P_s e c'_u}{I_1} = -980 + 1750 = +770 \text{ lb. per sq. in. (tension).}$$

$$f'_b = \frac{-P_s}{A_1} - \frac{P_s e c'_b}{I_1} = -980 - 1630 = -2610 \text{ lb. per sq. in. (compression).}$$

The tension in the upper fiber is close to the bond strength of the mortar, excellent as it is, and this suggests the desirability of placing some pre-stress upon the upper rod, which may, if desired, be released when the design loading is applied to the beam. The pre-stress tension on the steel was only 34,600 lb. It should have been carried much higher.

Step II. Loading Until the Initial Compressive Stress on the Bottom Fiber Was Released.

$y_2 = 6.73$ and $I_2 = 675$ in.^{4}

The moment required to relieve pre-stress on the bottom fiber,

$$M'_2 = \frac{f'_b I_2}{c''_b} = \frac{2610 \times 675}{5.27} = 334{,}000 \text{ in-lb.}$$

$$f''_s = \frac{334{,}000}{675} \times 4.27 \times 4.75 = 10{,}050 \text{ lb. per sq. in.}$$

Total steel unit stress = 49,250 lb. per sq. in. (tension).

$$f''_u = +770 - \frac{334{,}000}{675} \times 6.73 = -2560 \text{ lb. per sq. in. (compression).}$$

During this stage the stress on the upper ceramic fiber changes from tension to compression and a small additional load appears to be added to the tensile load on the lower steel.

Step III. Final Bending and Calculation of Ultimate Stresses.—Because of the change in modulus of elasticity of the ceramic material for higher loads and because only a rough assumption can be made as to the proper section to use in the calculation, the values obtained here in the third step can be relied upon only to give an indication of what happens at the ultimate failure of this beam. For this step, $y_3 = 4.6$ in. and $I_3 = 317$ in.^{4}

$$f_u = +f'_u - \frac{M'_2 c''_u}{I_2} - \frac{M_u - M'_2}{I_3 + c_u}$$

$$= +770 - 3330 - \frac{561{,}000 - 334{,}000}{317 + 6.4} = -5860 \text{ lb. per sq. in. (compression).}$$

$$f_s = 39{,}200 + 10{,}050 + \frac{561{,}000 - 334{,}000}{317 + 6.4} \times 4.75$$

$$= 71{,}050 \text{ lb. per sq. in. (tension).}$$

TABLE I.—DATA ON BEAMS.

Beam	Elongation of Rods, in.	Pre-Stress, lb. per sq. in.			Loading	Span, ft:in.	Load, lb., to give Deflection		External Maximum Load, lb.	External Maximum Moment, M, in-lb.	Final Fiber Stress, lb. per sq. in.		Maximum Moment, Pre-Stress, $\frac{M}{f'_s}$	Type of Failure
		Steel	Ceramic											
		Tension, f'_s	Top, f'_u	Bottom, f'_b			$\frac{1}{600}$ of Span Length	$\frac{1}{360}$ of Span Length			Steel, f_s	Ceramic, f_u		
B-1.........	0.18	73 400	−1500	−1500	$\frac{1}{3}$ points	5:0	13 400	.....	18 400	184 000	84 330	−4600	2.51	compression
T-1.........	0.066	22 300	−168	−930	midpoint	5:0	.....	.....	17 920	269 000	71 260	−7880	11.5	shear
T-2.........	0.039	13 100	−98	−550	midpoint	5:0	.....	.....	10 350	155 000	41 070	−4530	12.6	shear
T-3.........	0.047	15 900	−120	−670	midpoint	5:0	.....	.....	14 930	224 000	59 380	−6810	14.1	shear
T-4.........	0.22	51 100	−390	−2162	midpoint	9:0	3 000	5 500	10 790	291 000	77 240	−6040	5.7	compression
T-5.........	0.20	46 500	−345	−1965	midpoint	9:0	4 500	7 000	11 210	303 000	80 440	−6810	6.5	compression
L-1.........	0.20	36 900	+770	−2440	4 points[a]	12:4	13 300	19 500	29 300	561 000	71 050	−5860	15.20	diagonal tension
L-2.........	0.15	29 390	+580	−1960	4 points[a]	12:4	10 800	.....	18 000	423 000	53 290	−4400	15.17	diagonal tension

[a] The distances between the loading points are as follows:

	Between Support and First Point of Loading, in.	Between First and Second Points of Loading, in.	Between Second and Third Points of Loading, in.	Between Third and Fourth Points of Loading, in.	Between Fourth Point of Loading and Support, in.
L-1........	25.18	26.25	45.15	26.25	25.18
L-2........	36.40	18.80	37.60	18.80	36.40

Very evidently the strength of the lower steel was not completely utilized, indicating that the pre-stressing should have been carried much further. If this had been done the upper rod would also have been placed under tension to prevent the joints from opening at the top, and the calculations would have been somewhat changed.

During the third stage, the tensile load is transferred from the ceramic material to the steel as in any ordinary reinforced beam. In this particular beam the transfer apparently starts at a load of about 14,000 lb., as judged by the marked inflection of the "tension" curve plotted from the strain gage readings taken on the bottom fiber. The other curves have smaller inflections at this same loading and the bottom rods appeared to elongate much more rapidly beyond this point. During and after this transfer, the neutral axis rose again.

The results obtained, as well as experience with other types of pre-stressed members, indicate clearly the importance of the proper amount of pre-stressing. The data for several beams are given in Table I.

COLUMN CONSTRUCTION

The results obtained with the beams suggested the possibility of securing marked improvements over usual brick masonry columns which can carry only from 20 to 30 per cent of the compressive strength of the units. To take advantage of the properties of well-burned ceramic units it is proposed to:

1. Make the cross-section of the column of one single unit, taking advantage of the tensile strength of the ceramic unit and avoiding the disadvantages of steel hoops in the joints.
2. Use one, two or four rods, according to the size of the section, running lengthwise to aid in developing mortar strength and to be pre-stressed to place a few hundred pounds per square inch load on the column, in order to resist bending in the slender columns obtainable and to resist the splitting tendency at the ends of the beams noted below. A steel of high yield point would not be so important as in the beams.
3. Use the mortar mix and assembly methods described for beams.

Three columns were fabricated of special paving brick extruded with a hole in the middle for an assembly rod. In cross-section the brick were 3.75 by 7.75 in. One column was made from units 2.25 in. thick, another from units twice this dimension, and the third with brick 9 in. think; but all three had closely similar ultimate strengths. The columns were nearly 10 ft. long and were broken at the age of about 40 days, using plaster caps after cutting off the ends of the rods. As suggested above, it is believed now that the rod should have been pre-stressed slightly. Failure occurred by the units at one end, splitting and crushing at a load averaging 6000 lb. per sq. in., in spite of a length-thickness ratio of about 32. Bats of this brick, with plaster caps, were crushed under a load averaging slightly more than 12,000 lb. per sq. in., indicating a considerable increase in the ratio of column unit crushing strengths over that obtained with usual masonry.

CONCLUSIONS

1. Building members assembled by the methods described above have developed noteworthy stiffness, which seems to be dependent to a considerable extent upon the amount of pre-stressing.
2. The beams have shown excellent load-carrying capacity relative to their cross-sectional area and weight.
3. The ultimate bending moment seems to vary with the pre-stress.
4. Failure in the long beams with center loading was in compression while the shorter beams, or the longer beams under multiple loading failed in diagonal tension (or shear).

5. The stiffness of the members and the comparatively small amount of bending of the stress-strain curve should help to simplify the design of such beams.

6. Accurate control of the pre-stressing operation, readily secured, is very important.

7. Pre-stressing apparently causes a shift upward of the neutral axis. On loading the member this axis approaches the bottom ceramic fiber as the initial compressive pre-stress thereon is neutralized, probably followed by a rise again with additional loading.

8. Columns of remarkable load-carrying capacity, even when quite slender, have been fabricated.

9. Savings can be secured by reductions in weight and volume over present building members.

10. These members, properly protected at the ends, are fireproof and weatherproof, thereby increasing materially the savings.

Acknowledgments:

The tests on the tee and brick beams and on the columns were carried out in the Testing Materials Laboratory of the Carnegie Institute of Technology and grateful acknowledgment is made to Prof. F. M. McCullough for permission to use the testing machines.

The tests on the L beams were conducted by George W. Colton, of Yale University, to whom our thanks are also due, as well as to Earle T. Andrews, of Mapleton Depot, Pa., for valuable suggestions regarding the analysis.

MEASUREMENT OF PARTICLE SIZE DISTRIBUTION BY OPTICAL METHODS

By R. N. Traxler[1] and L. A. H. Baum[1]

Synopsis

Studies were made of the various factors influencing the determination of particle size distribution by means of the Wagner turbidimeter. A technic was developed for the dispersion of the finely pulverized solids. The results obtained on cement, trap rock, silica, limestone, and green, red, and black slate powders using the turbidimeter were compared with the size distribution data given by an air analyzer of the type described by Roller.

It was concluded that a direct proportionality does not exist between surface area (concentration of suspension) and turbidity. Further, turbidity is influenced by the particle size and optical properties of the dispersed solid. For the accurate investigation of particle-size distribution in mineral powders of the types here studied, the turbidimeter is apparently unreliable. However, for the detection of changes in size distribution in a single product the method is no doubt of practical value.

For some time an air analyzer of the type designed by Roller[2,3,4] has been used in our laboratory for the determination of the particle size distribution in commercially available mineral powders. The results obtained on a number of powders commonly used as fillers or stabilizing agents for bituminous materials have already been reported.[5,6] When properly conducted by an experienced operator, this direct method of analysis gives accurate information concerning the weight percentage of the various size fractions present in a powder, provided the mineral does not fall into class 1 on the scale of hardness. However, some equally accurate method, applicable also to very soft minerals, is desirable for obtaining a complete size distribution analysis in a few minutes instead of several hours.

Although turbidimeters have been used for the rapid estimation of average particle size, nevertheless few attempts have been made to apply such instruments to the determination of size distribution. Some time ago

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[1] Technical Bureau, Research Division, The Barber Asphalt Co., Maurer, N. J.

[2] P. S. Roller, "Separation and Size Distribution of Microscopic Particles. An Air Analyzer for Fine Powders," U. S. Bureau of Mines *Technologic Paper No. 490* (1931).

[3] P. S. Roller, "Accurate Air Separator for Fine Powders," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 3, p. 212 (1931).

[4] P. S. Roller, "Measurement of Particle Size with an Accurate Air Analyzer: The Fineness and Particle Size Distribution of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 607 (1932).

[5] J. S. Miller, Jr. and R. N. Traxler, "Fundamental Physical Properties of Mineral Fillers—I," Tenth Annual Asphalt Paving Conference, New Orleans, La., December 5–7, 1932, The Asphalt Inst., New York City.

[6] R. N. Traxler and L. A. H. Baum, "Determination of Particle Size Distribution in Mineral Powders by Air Elutriation," *Rock Products*, June, 1934, p. 44.

Stamm and Svedberg[1] employed the scattering of light to determine the distribution of particle sizes in emulsions. Recently Wagner[2] designed a turbidimeter by means of which he obtained data on the particle size distribution in portland cement. This method utilized a relationship between the degree of turbidity of a suspension and the surface area of the dispersed solid, together with Stokes' law predicting the rate of fall of spherical particles through a viscous medium.

A Wagner turbidimeter was obtained. Following the directions given for the operation of that apparatus, preliminary experiments with various mineral powders gave results differing markedly from those obtained with the air analyzer. Consequently, it seemed necessary to investigate the method more critically. Powders, samples of which had been carefully fractionated in the air analyzer, were used to determine the completeness of dispersion and the influence of concentration, particle size and the optical properties of the mineral on the results given by the turbidimeter. Each size fraction obtained from the air analyzer was examined under the microscope and the three axes of each of the particles in several fields were measured. From these values the harmonic mean diameter of each particle was calculated, the reciprocal of which is proportional to the specific surface of the particle. Knowing the weight of each fraction and having microscopically determined the surface mean diameter of each fraction[3] (which checked with the Stokes' law calculated mean) the total surface area per gram of the powder is known directly.

TABLE I.—PARTICLE SIZE DISTRIBUTION IN STANDARD CEMENT SAMPLE NO. 114.

Size Fraction, μ	Weight, per cent	
	Air Analyzer	Turbidimeter Using Kerosine – Oleic Acid
less than 5	18.0	14.4
5 to 10	14.7	15.8
10 to 20	20.6	22.6
20 to 40	29.0	29.4
40 to 80	17.7	17.8
Specific surface	2233 sq. cm. per g.	2142 sq. cm. per g.

Dispersion of Powder:

In his study of particle size distribution in cements, Wagner used a solution of oleic acid in kerosine as the dispersion medium for the powder. Such a solution and the motor-driven brush furnished with the turbidimeter were used to disperse a standard cement sample, No. 114, obtained from the National Bureau of Standards. Wagner's method of calculation and his assumed size for the largest particle passing a No. 325 sieve were employed. The value obtained for the specific surface of the cement checked closely with that given by the Bureau of Standards.

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[1] A. J. Stamm and T. Svedberg, "The Use of Scattered Light in the Determination of the Distribution of Size of Particles in Emulsions," *Journal*, Am. Chemical Soc., Vol. 47, p. 1582 (1925).

[2] L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

[3] P. S. Roller, "Separation and Size Distribution of Microscopic Particles. An Air Analyzer for Fine Powders," U. S. Bureau of Mines *Technologic Paper No. 490* (1931).

In addition, readings were made which included the minus 5 μ fraction and in which 45 μ was assumed to be the largest particle passing a No. 325 sieve. Such changes in procedure, made in order to obtain a direct comparison with the air analyzer results, necessitated slight modifications in Wagner's equation. The values obtained are recorded in Table I.

The size distribution in the standard cement sample No. 114 was then determined in the air analyzer. In the calculation of specific surface from the data obtained by means of the air analyzer, 2.5 μ was assumed as the surface mean diameter of the minus 5 μ fraction. Although the minus 5 μ fraction of the cement was not examined microscopically to determine the surface mean diameter, nevertheless from our experience with other powders it is believed that 2.5 μ may be too large and consequently the value for specific surface given in the second column of Table I may be too small. In all of the mineral powders discussed below the microscopically determined mean for the finest fraction is used.

A kerosine - oleic acid solution was then used to disperse several of the commercial mineral powders which had been fractionated previously by the air analyzer. Although a number of the suspensions were digested under a reflux condenser for 30 to 60 min. in an attempt to increase the wetting (dispersion) of the powders by the kerosine, the data on the kerosine - oleic acid suspensions would indicate that most of the powders were considerably coarser than shown by the measurements made by means of the analyzer.

Two explanations may be offered for the discrepancies between the results obtained by the two methods: (1) the air analyzer showed the powder to be too fine due to pulverization during the analysis, or (2) the turbidimeter indicated a coarser powder than actually existed due to poor dispersion or other reasons. The first explanation did not appear satisfactory because it had been proved by exhaustive tests in the air analyzer that comparatively little pulverization of hard minerals occurred during a properly conducted experiment. If pulverization does not occur in the air analyzer the rate at which a particular size fraction is blown over decreases abruptly when all of the particles of that size, originally present in the powder, have been removed. If grinding occurs in the air analyzer (as will happen with very soft minerals) no appreciable decrease in rate will occur because fine particles are produced almost as fast as they are removed. For all the minerals used in this investigation a sharp break occurred in the curve obtained from a logarithmic plot of grams of powder blown over per hour against time in minutes. Also, the fact that microscopic examination of the material blown over at the end of a particular fractionation showed all of the particles to be of maximum and uniform size was further proof that attrition did not occur.

Experience with the air analyzer had proved the necessity of obtaining complete dispersion of the small mineral particles. If the particles cling together and form aggregates in the suspension the data obtained will be worthless no matter how carefully the subsequent manipulations are conducted. Hence a thorough study was made concerning the preparation of suspensions for use in the turbidimeter.

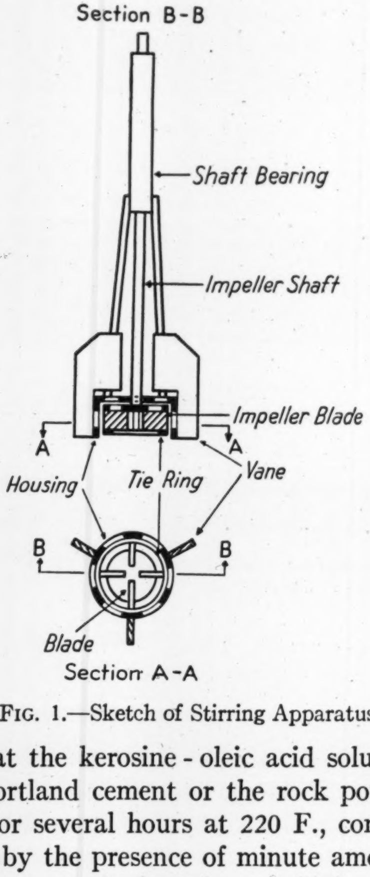

FIG. 1.—Sketch of Stirring Apparatus.

It is possible that the kerosine - oleic acid solution did not properly disperse either the portland cement or the rock powders. Although the powders were dried for several hours at 220 F., complete dispersion may have been prevented by the presence of minute amounts of moisture. A black slate powder was synthesized by combining weighed amounts of various size fractions previously separated by the air analyzer. Since the surface area of each fraction was calculated from the microscopically measured dimensions of the particle, the surface area of the mixture was known. This "synthetic" powder was dispersed in kerosine - oleic acid solution and the surface area determined by means of the turbidimeter. The value thus obtained for the specific surface of the powder was 1970 sq. cm. per g.

whereas the directly measured specific surface was 5124 sq. cm. per g. Evidently the results obtained on such a powder with the turbidimeter using kerosine - oleic acid as a dispersing medium are of no value.

Although water could not be used with the cement, a number of different aqueous solutions were employed as dispersing media for several rock dusts. Solutions of sodium oleate, Na_2CO_3, Na_3PO_4, saponin and "S" brand sodium silicate were tried and all found to give better dispersions than kerosine and oleic acid. After considerable experimentation, solutions containing 0.0125 per cent of saponin or 0.05 per cent of "S" brand silicate were found to give dispersions which showed the greatest surface area and consequently were the most satisfactory dispersing agents for the powders being investigated.

The rotating brush supplied with the turbidimeter to disperse the powder drew air into the liquid because of the vortex produced. In the saponin and "S" brand silicate solution the gas was so thoroughly dispersed that a measurable but variable turbidity resulted without the presence of any mineral. To avoid this difficulty a mechanical stirrer, illustrated in Fig. 1, was developed. When this agitator was placed below the surface of the liquid, powder and solution were thoroughly mixed without concomitant dispersion of air.

TABLE II.—PARTICLE SIZE DISTRIBUTION IN SILICA DUST BY THE TURBIDIMETER.

Size Fraction, μ	Weight, per cent		
	Oleic Acid in Kerosine	Aqueous Solutions of Saponin	Aqueous Solutions of "S" Sodium Silicate
less than 5	4.2	24.4	28.8
5 to 10	15.2	19.9	16.7
10 to 20	12.7	21.9	22.0
20 to 40	51.3	25.5	21.8
40 to 80	16.6	8.3	10.7

Table II gives a comparison of the particle size distribution by weight obtained on a sample of pulverized silica using the turbidimeter and different dispersion media. In the calculations account was taken of the differences in viscosity of the various solutions used in dispersing the powder.

Relationship Between Concentration of Suspension and Turbidity:

Wagner states[1] that cement dust dispersed in a kerosine - oleic acid solution gives a linear relationship between the concentration of the suspension and its turbidity expressed by $\log I_o - \log I_d$, where I_o is the intensity of the light transmitted through the dispersing medium and I_d is the intensity transmitted through the suspension. Since I_o is adjusted to 100 microamperes, log I_o is 2 and thus log I_o − log I_d may be written 2 − log I_d.

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[1] L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

In order to establish whether this relationship was valid for various minerals, each possessing characteristic optical properties, six different powders were analyzed in the turbidimeter using three or four concentrations of each material in aqueous saponin solution. Silica dust was also dispersed in kerosine - oleic acid. The improved stirring device was used in all of the experiments; Table III shows the data obtained.

TABLE III.—DATA SHOWING THE RELATIONSHIP BETWEEN CONCENTRATION OF SUSPENSION AND TURBIDITY.

Powder	Dispersing Medium	Weight, W, g. per liter	$2 - \log I_d$	$\frac{2 - \log I_d}{W}$
White silica........	kerosine – oleic acid	3.0	0.8697	0.289
		2.0	0.6383	0.319
		1.5	0.5086	0.339
		1.0	0.3516	0.352
White silica........	aqueous saponin solution	3.0	1.6198	0.539
		1.5	1.1805	0.787
		0.75	0.7305	0.975
		0.375	0.3925	1.048
White limestone...	aqueous saponin solution	2.5	1.6990	0.679
		1.5	1.3010	0.869
		1.0	1.0000	1.000
		0.6	0.6383	1.062
Gray trap rock.....	aqueous saponin solution	1.86	1.5530	0.836
		0.90	0.8539	0.948
		0.75	0.7258	0.970
		0.60	0.5607	0.930
Green slate........	aqueous saponin solution	2.0	1.8861	0.945
		1.0	1.3468	1.347
		0.66	1.0132	1.540
		0.60	0.9136	1.520
Black slate........	aqueous saponin solution	0.87	1.4437	1.660
		0.75	1.2000	1.600
		0.60	0.9586	1.590
		0.30	0.4300	1.432
Red slate..........	aqueous saponin solution	0.60	1.6021	2.670
		0.30	0.7212	2.400
		0.15	0.3466	2.320

In Fig. 2 concentration of suspension in grams per liter is plotted against the turbidity for unit concentration. If a direct proportionality existed and c was a constant, the lines in Fig. 2 would all be parallel to the concentration axis. However, since the curve for silica dust dispersed in kerosine - oleic acid is the only one which approaches such a condition it must be concluded that the relationship between turbidity or optical density and concentration of suspension is not a direct proportionality.

Thus, for the minerals studied, the equation $s = c\,(\log I_o - \log I_d)$ which relates surface and turbidity cannot be used to calculate accurately surface area, since the so-called transmittancy constant c is not a constant but varies with the concentration of the suspension (and also with particle size and optical properties as will be shown below). When the slopes of the curves given in Fig. 2 are negative as they are for silica, limestone, trap rock, and green slate dust, the values of c must become larger as the concentration increases. Conversely, if the slopes are positive as is true for black and red slate c decreases as the concentration increases.

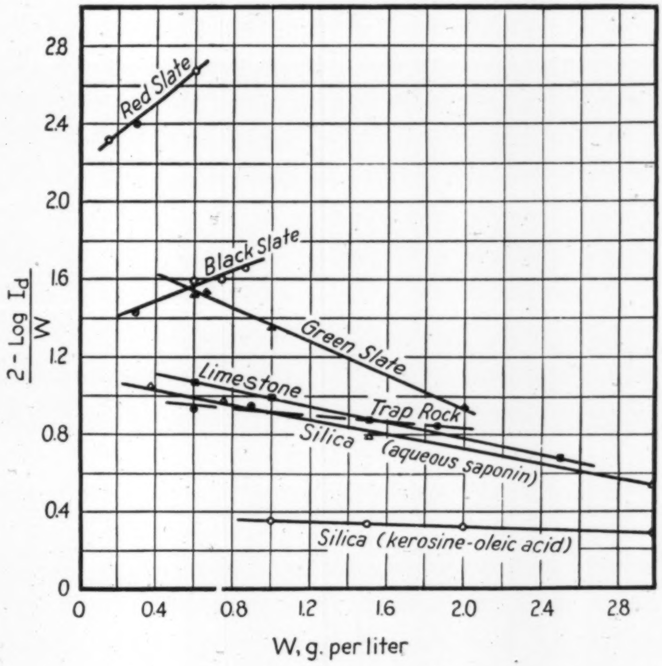

FIG. 2.—Turbidity-Concentration Curves.

The differences in the relationships between turbidity and concentration noted for the various colored minerals may be due to their selective absorption of light.[1] For example, the green slate absorbs the long wave lengths and permits the green light to reach the photoelectric cell, whereas the red slate absorbs the short wave lengths and permits the red light to pass. After determining the sensitivity of the photoelectric cell to various wave lengths experiments should be conducted using different kinds of monochromatic light with the various powders.

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[1] G. F. A. Stutz and A. H. Pfund, "A Relative Method for Determining Particle Size of Pigments," *Industrial and Engineering Chemistry*, Vol. 19, p. 51 (1927).

Variation of Transmittancy Constant c with Particle Size:

Since there were available carefully size-graded fractions of different powders whose surface mean diameters had been carefully determined by microscopic measurements of the three dimensions of the particles, it was possible to determine the transmittancy constant *c* directly for different size fractions. The various size fractions were dispersed in saponin solution using the improved technic. None of the fractions settled faster than predicted by Stokes' law, consequently, it would appear that all of the powders were completely dispersed. Table IV gives the values for the transmittancy constant *c* for various size fractions of white silica and green slate powder.

The differences in the value of *c* for the various fractions, shown in Table IV, may be due in part to the different concentrations of suspension necessarily employed because of the limited range of light intensity capable of being measured in the turbidimeter. However, it appears that the relationship between transmission of light and surface area of suspended material is dependent on particle size and that the value of *c* becomes smaller as the particle size increases. Essentially the same conclusion was reached by Lowry and Marsh[1] in their study of size fractions of barytes suspended in medicinal paraffin.

TABLE IV.—VALUES FOR TRANSMITTANCY CONSTANT *c*.

Size Fraction, μ	Transmittancy Constant "c"	
	White Silica	Green Slate
less than 2	2580	1680
2 to 5	....	819
5 to 10	1290	722
10 to 20	....	550
20 to 40	955	476

Incorrect Assumptions Concerning Maximum Particle Size Measured by the Turbidimeter:

Changes in turbidity caused by large particles falling from suspension are so small that it is customary to analyze the fractions retained on a No. 325 sieve by means of sieves. The largest particle passing a No. 325 sieve is usually assumed to have an average diameter of about 60 μ because a square opening measuring 43 μ on a side has a diagonal of about 60 μ. If only two dimensions (length and breadth) of particles just passing this sieve are measured by means of a microscope the average diameter usually appears to be 60 μ. However, by measuring the three axes of a large number of particles just passing a No. 325 sieve it was found that the average diameter is nearer 45 μ.

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[1] T. M. Lowry and M. C. Marsh, "Properties of Powders. Part IX. The Scattering of Light by Graded Particles in Suspension," *Transactions*, Faraday Soc., Vol. 24, p. 195 (1928).

TABLE V.—SILICA DUST DISPERSED IN AQUEOUS "S" BRAND SODIUM SILICATE.

Size Fraction, μ	Weight, per cent	
	60 μ, max.	45 μ, max.
less than 5	26.5	28.8
5 to 10	15.9	16.7
10 to 20	20.9	22.0
20 to 40	20.8	21.8
40 to 80	15.9	10.7

TABLE VI.—COMPARISON OF SIZE DISTRIBUTION AND SPECIFIC SURFACE DATA.

Powder	Size Fraction, μ	Composition by Weight, per cent	
		Air Analyzer	Turbidimeter
Silica	less than 5	23.7	24.4
	5 to 10	12.7	19.9
	10 to 20	22.9	21.9
	20 to 40	31.0	25.5
	40 to 80	9.7	8.3
	Specific surface	4352 sq. cm. per g.	3520 sq. cm. per g.
Limestone	less than 5	19.7	23.2
	5 to 10	12.7	14.1
	10 to 20	15.9	16.0
	20 to 40	25.0	20.0
	40 to 80	20.1	18.9
	over 80	6.6	7.8
	Specific surface	3520 sq. cm. per g.	3019 sq. cm. per g.
Trap rock	less than 5	20.9	12.6
	5 to 10	16.0	16.0
	10 to 20	21.3	21.9
	20 to 40	21.4	26.2
	40 to 80	16.4	19.3
	over 80	4.0	4.0
	Specific surface	3241 sq. cm. per g.	1935 sq. cm. per g.
Green slate	less than 5	41.9	28.0
	5 to 10	15.0	19.3
	10 to 20	10.9	19.9
	20 to 40	19.7	22.7
	40 to 80	12.0	9.6
	over 80	0.5	0.5
	Specific surface	6235 sq. cm. per g.	3662 sq. cm. per g.
Black slate	less than 5	31.2	21.3
	5 to 10	22.5	26.0
	10 to 20	27.5	30.3
	20 to 40	17.5	21.1
	40 to 80	1.3	1.3
	Specific surface	5124 sq. cm. per g.	3037 sq. cm. per g.
Red slate	less than 5	41.7	29.0
	5 to 10	18.3	15.3
	10 to 20	16.8	23.1
	20 to 40	19.0	24.0
	40 to 80	4.2	8.6
	Specific surface	7840 sq. cm. per g.	4140 sq. cm. per g.

In all the tests run on rock powders the average diameter of the largest particle passing a No. 325 sieve has been assumed to be 45 μ. Table V shows the difference in particle size distribution in silica dust when 60 and 45 μ are assumed as the size of the largest particles passing this sieve.

Comparison of Data Obtained by the Air Analyzer and the Wagner Turbidimeter:

In Table VI a comparison of the size distribution and specific surface data obtained on six different mineral powders by means of the air analyzer and the turbidimeter are given. The weights in per cent of the various fractions were obtained directly with the air analyzer whereas those with the turbidimeter were obtained indirectly by calculations from the surface area. In the calculation of specific surface from the air analyzer data the microscopically determined surface mean diameter for the minus 5 μ fraction was used. This value was always less than 2.5 μ. For the larger fractions the Stokes' law averages (7.5 μ for the 5 to 10 μ fraction) were employed.

If 2.5 μ had been used in calculating the surface area of the minus 5 μ fraction the specific surface of the powder would have been much lower. For example, such an assumed surface mean diameter for the minus 5 μ fraction of silica dust would result in a specific surface for the whole powder of 3180 sq. cm. per g. instead of 4352 sq. cm. per g. (Table VI) calculated from the microscopically determined surface mean diameter of 1.6 μ for the finest fraction.

Since the value for the surface mean diameter of the finest fraction is often arbitrarily selected, the data frequently given for surface area are unsatisfactory for estimating the fineness of powders. Perhaps a more satisfactory constant for evaluating fineness is the volume mean diameter which weights or emphasizes the finer fractions to a smaller degree. Any assumption or error made concerning the average particle size of the fine fraction introduces a smaller error into the value for the volume mean diameter of the whole powder than that which occurs when the surface mean diameter is determined. Further, as indicated by unpublished data from this laboratory, the volume mean diameter of a powder is more useful than the surface mean in explaining the flow properties of a viscous system composed of solid particles dispersed in a liquid.

For any powder the reading with the turbidimeter taken before any settlement occurred allowed the calculation of the specific surface of the unfractionated powder. Successive readings gave the surface areas of the larger fractions and from these the weights in per cent were calculated using the Stokes' law average diameters for the various fractions. Readings were made down to 5 μ and 45 μ was taken as the size of the largest particle passing a No. 325 sieve. These changes from Wagner's procedure necessitated slight alterations in his equations.[1] The surface area and the weights in per cent of the minus 5 μ fraction were determined by difference.

In this work aqueous saponin solutions and the improved agitator illustrated in Fig. 1 were used in preparing the dispersions for the turbidimeter.

The discrepancies between the air analyzer and the turbidimeter are due presumably to the fact that the relationship between surface area and turbidity is not a direct proportionality and is influenced by the concentration of suspension, the particle size, and optical properties of the dispersed solid.

As a general research instrument for the accurate determination of particle-size distribution in mineral powders of various types the turbidimeter is apparently unreliable because of the specific character of each mineral. For the detection of changes in size distribution in a single product the method is no doubt satisfactory.

Acknowledgment:

The authors are indebted to Mr. C. U. Pittman for valuable suggestions and criticisms during the preparation of the manuscript, and to Mr. H. E. Schweyer for the drawings.

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[1] L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

DISCUSSION

Mr. P. S. Roller[1] (*presented in written form*).—An interesting feature of this paper is the consideration given to the size measurement of each particle. Instead of the customary examination merely of the superficial dimensions, the authors have considered also the depth of the particle. This point is important in connection with the use of the air analyzer. On the "break" in the rate-time curve and near the "end point" of a fractionation, a fairly uniform aggregate of particles presents itself. The mean size of this aggregate determines the location of the end point. If the mean size is estimated with reference to the superficial dimensions only, the end point will be incorrect and will precede the true end point determined from measurement of the three dimensions of each particle by about 2 to 4 per cent. Originally[2] the writer considered the end point on the basis of the superficial dimensions only, with the result that the end point was taken somewhat short.

Indirect methods of determining particle size, that is methods which do not involve a physical separation of the required sizes,[3] are doubtless useful under many conditions. Such indirect methods will practically always give results that vary in the same way as the fineness. However, when definite assurance is desired that the size values given by the indirect method are real and not merely relative, the results should be confirmed by some direct method of analysis. The need for such confirmation is indicated particularly when passing from one material to another, as the properties of the new material may be such as to weaken seriously the correctness of the assumptions made in the indirect method even though these assumptions may be correct for the original material. With the new material, as with the old, the property measured will vary usually in the same direction as the fineness, but one cannot be sure that the derived quantity is the true size distribution. At present, the true size distribution probably is approached most closely by an accurate fractionation, fortified, at least in the first instance, by microscopic examination and check of the fractions. The paper of Traxler and Baum is valuable for the detailed manner in which this question has been treated in the case of optical measurement of size distribution.

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[1] Associate Chemist, U. S. Bureau of Mines, Nonmetallic Minerals Experiment Station, New Brunswick, N. J.

[2] P. S. Roller, "Measurement of Particle Size with an Accurate Air Analyzer: The Fineness and Particle Size Distribution of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 607 (1932).

[3] P. S. Roller, "Separation and Size Distribution of Microscopic Particles—An Air Analyzer for Fine Powders," U. S. Bureau of Mines *Technologic Paper No. 490* (1931).

MR. L. A. WAGNER[1] (*presented in written form*).—In this paper reference is made to two devices as representing means by which accurate measurements of the surface area of powdered materials may be obtained, namely, the microscope and the Roller air elutriation apparatus. For the benefit of those not experienced in the size measurement of small particles, it is well to point out again that the true surface areas of materials such as portland cement or powdered rock remain today quite unknown. To verify this statement we need only to remember that at the start of all particle size work it is necessary to assume that the particles have some regular shape. It is generally assumed that they are spheres and their size is expressed as some diameter. Actually, however, the particles are infinitely irregular in shape. From the first assumption on, it is obvious that any intimation that accurate values of surface are being measured is false. This first assumption must be followed by others depending upon the method of measurement being used. The resulting values are always arbitrary and only have meaning when defined in terms of the assumptions, apparatus, and method by which they were derived. The selection of any one apparatus, method, and accompanying set of assumptions as the means of obtaining a true measurement of the size and surface of pulverent material is still open to question.

In this connection, since the authors have obtained with the microscope what they consider an accurate knowledge of the total surface area of these powders, I should like to have them describe what means they used to measure the particles in the smallest fraction which cannot be seen at all with the microscope but which reflect light when subjected to dark ground illumination.

The authors found the transmittancy constant c to vary with particle size. I also found this to be the case and so did Alexander Klein in a turbidimeter paper reported before this Society last year.[2] I determined the extent of this variation for portland cement and included it in the calculation of surface area. This procedure increased the amount of computation for each determination and since it was desired that the operation of the turbidimeter be rapid, c was assumed to be independent of particle size and the extra computation was omitted. In view of the fact that the final results are arbitrary in any case, this short cut is felt to be justified.

A more serious matter, however, is the finding that the transmittancy constant varied with concentration of the suspension for all the powders examined except *white silica in kerosine*. Fortunately, the authors have presented enough of their data so that we can deduce why they encountered this variation in c and also how it may be avoided. In brief, too much powder was used in preparing the suspensions and with such dense concentrations the direct proportionality between weight of sample and turbidity broke down. Users of the turbidimeter quite generally recognize that suspensions which result in reading less than 10 microamperes ($2 - \log I_d$ greater than unity) are too dense to give reliable results. Such suspensions are discarded and replaced with suspensions containing less weight of sample.

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[1] Assistant Chemical Engineer, Cement Reference Laboratory, National Bureau of Standards, Washington, D. C.

[2] Alexander Klein, "A Suspension Turbidimeter for Determination of Specific Surface of Granular Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 303 (1934).

Turning to Table III of the paper, fourth column, it is seen that the only powder having all suspensions in the region of proper concentration ($2 - \log I_d$ less than unity) *was white silica in kerosine.* Moving down the same column to gray trap rock, of the four suspensions reported, three are in the region of proper concentration, and for these three suspensions c is again constant enough for practical purposes.

I feel almost certain that if the authors will repeat this work using suspensions in the concentration range suggested above they will find c to be constant and the instrument will no longer be unreliable.

MR. R. N. TRAXLER.[1]—The criticism expressed in the first paragraph of Mr. Wagner's discussion is certainly true for any method which cannot be checked by *direct* observation. Microscopic examination of the closely limited size fractions obtained by the Roller air analyzer proves that the application of Stokes' law to irregularly shaped particles is valid if the diameters of the particles are defined in terms of the ratio of surface to volume, or in other words, if the harmonic mean diameter is employed. Mr. Roller has emphasized this point in his discussion. Of course it must be admitted that even after such direct measurement and correlation there probably is still some error in the estimation of surface area due to secondary shape irregularities. This error is negligibly small compared to the errors encountered in indirect methods which do not involve separation and actual measurement of the particles.

We employed a projection apparatus attached to our microscope to aid in the measurement of the finer fractions. There were, of course, some very small particles which could not be seen even by that means, but they accounted for a very small percentage of the total surface area. However, if we had been able to see and count them they would have increased somewhat the surface area reported for the air analyzer data, thereby increasing the disparity between the results obtained by the two methods of analysis.

Mr. Wagner says that the effect of particle size on the transmittancy constant c may be ignored because the final results are arbitrary. The fact that the method is arbitrary is not a justification in itself for omitting a variable which influences the results. In this work we were interested in determining just to what extent an arbitrary result was obtained. Actually, as the data have shown, the arbitrariness is reflected in an unusually large divergence of the values of surface area from values that we regard to be close to the truth.

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[1] **Technical Bureau, Research Division, The Barber Asphalt Co., Maurer, N. J.**

The argument that the concentration of the suspension should be kept low in order that the values of $2 - \log I_d$ will be less than unity is justifiable. However, the data given in Table III and shown graphically in Fig. 2 were purposely obtained over a rather wide range of turbidity in a deliberate effort to determine the relationship between turbidity and concentration of suspension. The determinations with the turbidimeter recorded in Table VI, where the two methods of analysis are compared, were all made using suspensions so dilute that the values of $2 - \log I_d$ were less than unity.

In conclusion, it would greatly increase the accuracy and usefulness of the turbidimeter if it were equipped with a 100 milliameter, instead of a 50 milliameter.

A PENETRATION METHOD OF MEASURING SOIL RESISTANCE

By W. S. HOUSEL[1]

SYNOPSIS

The penetration measurements have been developed in response to a demand for a simple and inexpensive test for determining the properties of clay soils. Equipment, consisting essentially of a falling weight and a device for recording penetration per blow of a core sampling pipe, has been developed and standardized. The penetration data are thus obtained as a part of the routine sampling of soil and correlated with physical tests performed in the laboratory.

The results of approximately eight years of research have shown that penetration measurements follow a consistent law. The resistance to penetration has been defined in terms of a penetration index, which is the number of standard blows necessary to drive a 1¼-in. standard core barrel 6 in. A rational formula has been developed which expresses the resistance to penetration in terms of shearing resistance or actual cohesion of the soil. After analysis of the data, the results were correlated with independent tests of shearing resistance and with series of bearing capacity tests conducted as another phase of this investigation. The results of the comparison of such independent methods of measuring soil resistance show good agreement and indicate that measurement of physical properties of soil may be accomplished with reasonable accuracy.

The penetration method has been used to determine the bearing value of soil for both shallow and deep foundations and may be operated in a 12-in. casing to any desired depth. It has also been used to measure cohesion of soil as affecting tunnel construction in the deep underground and in connection with dredging operations. Its application has only been extended to cohesive soils such as clay as the method is not designed for granular materials. It appears that it would be a practical field test for measuring the bearing value of clay subgrade soils, being particularly applicable to those plastic soils which are the source of some difficulty in highway construction.

INTRODUCTION

The development of a penetration method of measuring soil resistance herein described was initiated in 1927 and has been in progress up to the present time. Research on this particular method is a part of a more comprehensive study of soil properties that is being conducted as a cooperative investigation by the University of Michigan, the Michigan State Highway Department and a number of engineering organizations both municipal and corporate in the City of Detroit.[2] In addition to penetration measurements the investigation has included a number of series of large-size bearing-capacity tests and measurements of earth pressure on underground structures.

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[1] Research Consultant, Michigan State Highway Dept.; and Assistant Professor of Civil Engineering, University of Michigan, Ann Arbor, Mich.

[2] Other organizations cooperating in the investigation of soil properties include the Wayne County Road Commission; Detroit Edison Co.; Department of Buildings and Safety Engineering; Department of Public Works and Rapid Transit Commission.

The objective of the studies of the penetration method is to correlate such measurements with soil behavior under practical conditions as observed in connection with the various construction projects which involve the physical properties of soil. Particular emphasis has been placed on the correlation of resistance to penetration with the bearing value of cohesive soils as measured by bearing-capacity tests and observed in the settlement of structures. The data so far available cover cohesive soils varying from very soft clay to materials which approach the characteristics of clay pan or clay hard pan. The method has been developed in connection with core sampling, recognizing that the penetration measurements may be combined with common procedure in obtaining soil samples by driving a core barrel into the soil which, when removed, contains a relatively undisturbed sample of the material. The samples thus obtained are used for

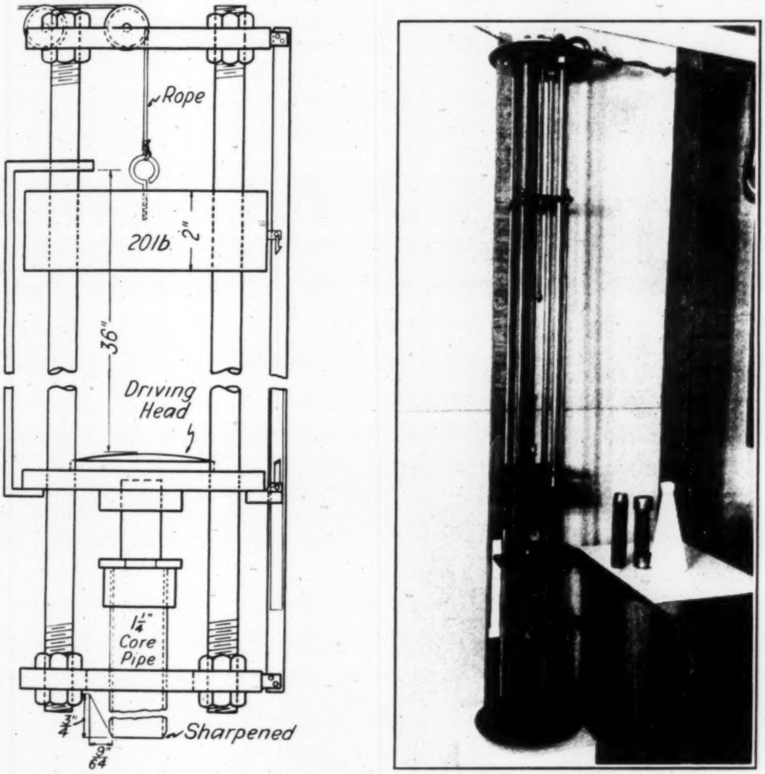


FIG. 1.—Penetration Apparatus.

various physical tests conducted in the laboratory and provide another source of data which may be used in the general correlation of soil properties.

Measurements of resistance to penetration are by no means new in the study of materials. In the case of soils, sounding with a rod, or the driving of a rod or pipe into the soil have been frequently employed by engineers and contractors as a means of estimating soil properties and as a rule-of-thumb method of correlating their general experience with soil behavior. With one or two exceptions within the author's knowledge there has been, however, no continued and consistent effort to develop accurate criteria by standardizing apparatus, correlating comprehensive data, and eliminating the personal equation involved in the loosely compiled empirical methods. The present investigation was undertaken specifically to fill this gap, as it appeared to be a promising field or investigation and one which might produce a dependable method of measurement. At the same time the method, if successfully developed, appeared to be sufficiently rapid and inexpensive to increase greatly the opportunity for the application of scientific methods of soil mechanics to practical problems.

DESCRIPTION OF TESTING APPARATUS

The device used in core sampling and observing penetration is shown in Fig. 1, and is simply a miniature drop hammer. It consists of four vertical steel rods which act as guides for a falling weight of 20 lb. The height of fall has been standardized as 34 in., being regulated by a stop which has later been changed to an automatic tripping device. The weight is raised by hand and at the bottom of the fall strikes the driving head to which the core-sampling pipe is attached. The penetration per blow is recorded on a carbon ribbon by a plunger attached to the driving head which is snapped against the carbon ribbon by a tripping device on the falling weight. The tripping device operates only when the weight is being raised and so records the position of the driving head and core sampling pipe after each blow. Figure 1 shows the device for recording the penetration per blow and typical examples of the ribbon on which the measurements have been recorded. The sampling pipe is also shown, illustrating the method of sealing the samples with small seamless cans and paraffin for shipment to the laboratory. The weight of the driving head, core-sampling pipe, and attachments has been standardized at 20 lb. The dead weight of the driving head absorbs part of the momentum of each blow due to inertia forces and must be considered in evaluating the force resisting penetration. The weight of the sampling pipe is usually variable so it is necessary to reduce the penetration to the basis of standard observations by a proper adjustment of the momentum absorbed by the dead weight of the moving parts.

The standard core sampling pipe or core barrel is a $1\frac{1}{4}$-in. steel pipe sharpened to minimize point resistance and emphasize side shear or frictional resistance. The pipe is sharpened on the outside to cut a core whose diameter is equal to the inside diameter of the pipe and to force the volume displacement equivalent to the volume of the wall of the core barrel toward the outside. The object in this is to eliminate squeezing action on the core in order to obtain a sample as little disturbed as possible. The primary reasons for minimizing point resistance are to avoid dimensional effects which cause a variation in the resistance to volume displacement under a loaded area, and dynamic effects particularly evident in plastic materials due to their well-known property of furnishing relatively high resistance to rapid displacement.

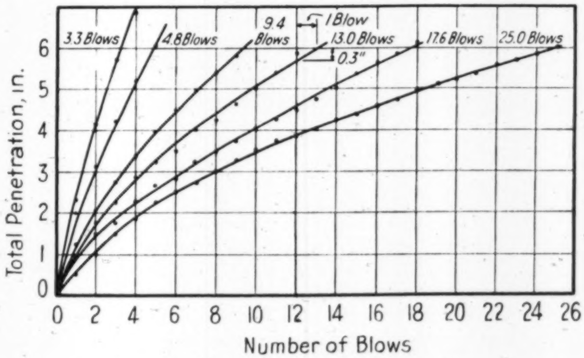


FIG. 2.—Typical Penetration Curves.

The sampling apparatus has been used to make penetration measurements and to obtain core samples to a depth of 80 ft. below the surface of the ground and has performed satisfactorily under such conditions. The sampling has been conducted in a 12-in. auger hole, the hole being kept open by driving a 12-in. steel casing, preferably following the sampling operation. The apparatus has been successfully used in an open auger hole to depths of 30 or 40 ft. When used in this way the apparatus is lowered to the bottom of the test hole, being kept vertical by circular liner plates at each end, and operated from the surface of the ground.

ANALYSIS OF PENETRATION MEASUREMENTS

Penetration Data:

The results of the penetration measurements are first plotted as illustrated in Fig. 2, with the penetration recorded after each blow of the standard weight plotted against the number of blows. Six-inch core samples have been used as a standard and the number of blows required for a penetration of 6 in. has been defined as the *penetration index.*

The penetration curves are used for two purposes; first to determine the number of blows and fractional parts of a blow for exactly 6-in. penetration, and second to facilitate the determination of the penetration per blow at various stages of driving. It is only in exceptional cases that penetration of 6 in. occurs at an even number of blows and the determination of fractional parts of a blow is the first refinement of the penetration data. In Fig. 2 are shown the penetration curves for soils which represent a range in the penetration index varying from 3.3 to 25 standard blows.

The slope of the penetration curves in Fig. 2 is measured graphically at penetrations of 2, 3, 4, 5 and 6 in. to determine the penetration per blow for substitution in the formula for the force resisting penetration, which must be expressed in terms of momentum absorbed for each blow of the falling weight. These slopes establish a relation between penetration per blow and the penetration index. The results of this procedure are illustrated in Fig. 3 which shows the relation between penetration per blow at 6 in. and the penetration index in blows. The data shown cover a number of investigations and a range in penetration index from less than 2 blows to 50 blows.

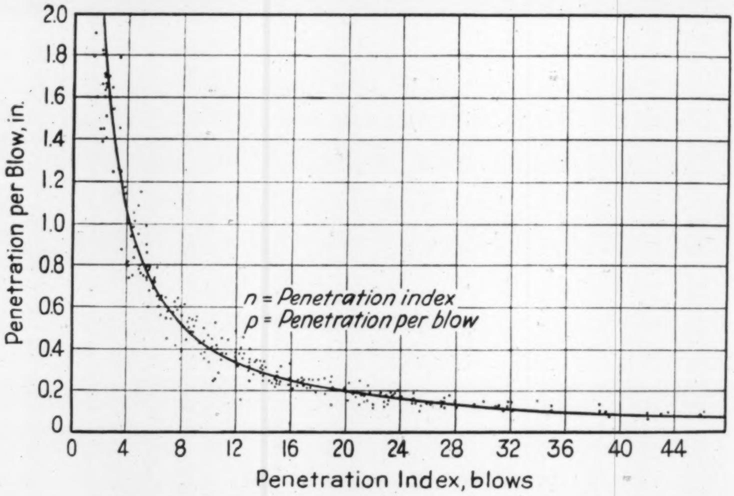


FIG. 3.—Penetration per Blow at 6-in. Penetration.

In Fig. 3 there is evident a consistent relation between the variables under consideration, and the curve and equation selected by graphical methods appears to represent this relation within the limits of experimental error involved in the data. The dispersion of the results of individual observations is no greater than might be anticipated but is great enough to indicate the necessity of some logical method of leveling the individual measurements before use in further analysis. For this reason, one variable, the penetration index, has been selected as the more reliable observation and the penetration per blow is determined by reference to the mean curve (Fig. 3) through the group of plotted points. It appears that the number of blows for a penetration of 6 in. may be observed with greater accuracy than the penetration per blow which depends upon measuring the slope of penetration curves and which also reflects irregularities in soil structure to a greater degree than the penetration index. Thus the penetration per blow from individual samples is leveled graphically as illustrated in Fig. 3 for any specific value of the penetration index or the same result may be computed from the equation of the curve.

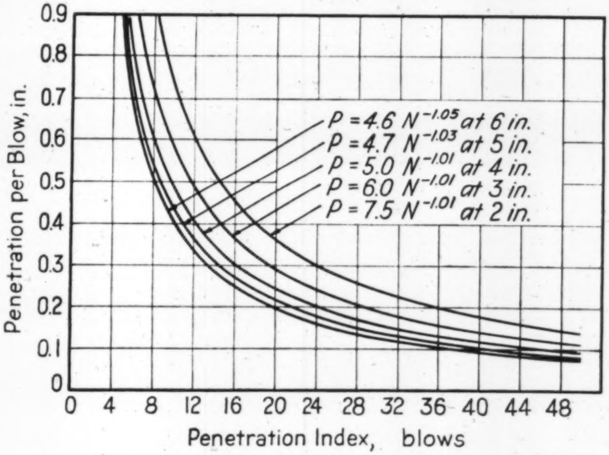


FIG. 4.—Penetration per Blow at Various Penetrations.

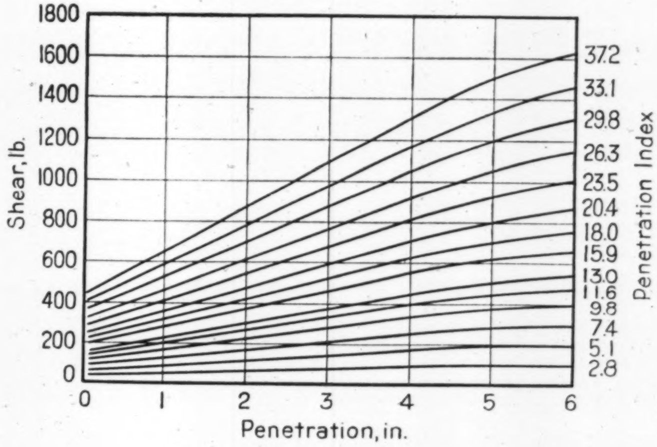


FIG. 5.—Typical Shear Curves.

Similar methods are used to determine the leveled penetration per blow at penetrations of 2, 3, 4 and 5 in. The group of curves shown in Fig. 4 with their respective equations were determined in the same manner as described above for 6-in. penetration.

In every case data from a number of investigations have been combined and the penetration per blow at various depths of penetration has been as consistent as that shown for 6-in. penetration. It should be noted that the curves and equations developed are representative of penetration measurements with the particular device used in this investigation and would not be valid for penetration measurements with different sizes of core barrels or with other modifications of apparatus. As will be evident from the later discussion of resistance to penetration, certain factors may be varied without changing the net result or invalidating the empirical relations used for the purpose of leveling data. No attempt, however, should be made to use these relations for penetration measurements under a different set of standards without thorough investigation substantiated by ample experimental evidence.

Resistance to Penetration:

The force resisting penetration may be evaluated in terms of the principle of the conservation of momentum by considering the transfer of the momentum of the falling weight to momentum of the core barrel and driving head which in turn is absorbed as the impulse of the force resisting penetration acting through a specific period of time.

$$\text{Momentum of falling weight} = Mv = \frac{W}{g}\sqrt{2gh}$$

where W = falling weight in pounds,
g = acceleration of gravity, and
h = height of fall in inches.

Let w be the weight of the driving head and pipe and v' be the maximum velocity of the dead weight. For a time period t_1 after impact the velocity of W changes from v to v' and the velocity of w changes from zero to v'. During a time period t_2 the velocity of $(W + w)$ changes from v' to zero. For small penetrations the force F resisting penetration and developed by the frictional or shearing resistance of the soil may be considered as a constant or average force. The momentum before impact must be equal to the momentum at any instant after impact plus the impulse exerted in overcoming the force F acting through a time t. This equality of impulse and momentum may be expressed for time periods t_1 and t_2 as follows:

$$\frac{W}{g}\sqrt{2gh} = \frac{W + w}{g} v' + Ft_1$$

$$\frac{W + w}{g} v' = Ft_2$$

$$\frac{W}{g}\sqrt{2gh} = Ft_1 + Ft_2 = Ft$$

The average velocity v_1 for time t_1 is equal to $v'/2$ and is also equal to the average velocity v_2 for time t_2 from which it follows that the time period

t_1 is equal to the time t_2 provided the average force F is the same for both time periods. Let p be the total penetration, then the total time t may be written:

$$t = \frac{p}{v_1} = \frac{2p}{v'}$$

$$Ft_1 = Ft_2$$

$$\frac{W}{g}\sqrt{2gh} = \frac{2(W + w)}{g} v'$$

$$v' = \frac{W}{(W + w)2}\sqrt{2gh}$$

$$t = \frac{4p\ (W + w)}{W\sqrt{2gh}}$$

$$Ft = F\,\frac{4p\ (W + w)}{W\sqrt{2gh}} = \frac{W}{g}\sqrt{2gh}$$

$$F = \frac{W^2\,2gh}{4pg\ (W + w)} = \frac{hW^2}{2p\ (W + w)} \quad \text{.......................................(1)}$$

In the equation for the force resisting penetration the various quantities are standardized as follows: $h = 34$ in.; $W = 20$ lb.; and $w = 20$ lb.

The penetration per blow, measured in the manner described is designated in the formula by p. When the dead weight w is more or less than the standard 20 lb., the force resisting penetration must be reduced to terms of this standard weight. The measured force F must not be altered and this condition requires that the product $2p(W + w)$ remain unchanged but that the two factors be adjusted to express penetration per blow as if the factor $(W + w)$ were 40 lb. The penetration per blow for a variable weight factor may be designated as p' and may be corrected for a constant value of $(W + w)$ of 40 lb. as follows:

$$2p'\ (W + w) = 2p \times 40$$

$$p = p'\,\frac{(W + w)}{40}$$

The equivalent penetration represents the penetration per blow for a constant dead weight and is used to determine the equivalent number of blows for 6-in. penetration or the penetration index from the curve or equation designated on Figs. 3 and 4.

Determination of Shearing Resistance:

The equation for F, the force resisting penetration, may be used to evaluate the shearing resistance of the soil. In Fig. 5 is shown a group of typical shear curves computed from the equation. The force F developed for penetrations from 2 to 6 in. has been plotted against the penetration in inches. The penetration per blow for each inch of penetration has been taken from the respective curves on Fig. 4 for each specific value of the penetration index. While each shear curve represents the soil resistance for an actual observation, the original data have been leveled in the manner previously outlined and each value of the penetration index has been reduced to terms of a constant dead weight of sampling apparatus. A large number of shear curves have been plotted using the original data and show the same consistent variation represented in Fig. 5 save for the usual range of variation in individual observations.

The slope of the shear curves represents the shearing force developed by the soil in pounds per inch of penetration or pounds per square inch of contact surface of the core sampling pipe multiplied by the inside and outside circumference of the pipe. The total force at zero inches penetration represents the initial resistance which may be designated as point resistance consisting of punching shear and resistance to volume displacement. The deviation of the shear curves from a straight line at higher penetration is evidence of the total shear on the core approaching a maximum value when no more soil is being forced into the sampling pipe. When the total shear developed on the core inside the pipe exceeds the resistance to volume displacement developed by the soil, continued driving of the sampling pipe only forces the soil aside and the length of the core remains the same. Repeated experiment with the standard sampling apparatus has shown that with the standard weight and height of fall used it is not feasible to obtain a greater length of core than 6 in. in the ordinary cohesive soils.

The resistance to penetration that has been measured is indicative of the true shearing resistance of the soil. Considerable evidence has been found which shows that in cohesive and plastic soils the force of adhesion between the soil and a foreign body driven into the soil is greater than the cohesive resistance of the soil itself. Piles which have been driven into the soil and pulled retain a coating or shell of soil around the contact surface. Observations of a large number of pile tests conducted for the purpose of measuring what may be termed shearing resistance indicate that the slipping occurs on a surface in the soil surrounding the pile, rather than an actual slipping between the pile and soil. The consistent variation between the penetration index and soil resistance as measured by independent tests indicates that the resistance to penetration is a measure of shearing resistance in the usual sense, rather than frictional resistance as commonly conceived.

The next step in the correlation is to establish the relationship between the shearing resistance developed by the soil and the penetration index.

The shearing resistance in pounds per square inch is obtained from the slope of the shear curves for different values of the penetration index shown in Fig. 5. Only the straight-line portion of the shear curve for each value of the penetration index has been used in the subsequent analysis inasmuch as no satisfactory measure of the shear area is available for penetrations greater than approximately 4 in. An analysis of the shear curves shows that they have a common point of intersection with the horizontal axis at a negative ordinate equivalent to a penetration of 2 in. The most convenient method of expressing the slope of the various curves is in terms of this hypothetical point of zero shear which amounts to nothing more than expressing the initial point resistance in terms of side shear.

The standard core-sampling pipe has an outside circumference of 5.22 in. and an inside circumference of 4.33 in. The shear area per inch of penetration is then 9.55 sq. in. The force resisting penetration as given by Eq. 1 may be expressed in terms of the penetration index as shown in the following demonstration:

Substituting $H = 34$ in., $W = 20$ lb., $w = 20$ lb. in Eq. 1, we have:

$$F = \frac{34 \times 20^2}{2 \times 40p} = \frac{170}{p}$$

At 2-in. penetration (see Fig. 4):

$$p = 7.5\ N^{-1.01} \qquad F = 22.67\ N^{1.01}$$

at 3-in. penetration:

$$p = 6.0\ N^{-1.01} \qquad F = 28.33\ N^{1.01}$$

at 4-in. penetration:

$$p = 5.0\ N^{-1.01} \qquad F = 34.00\ N^{1.01}$$

Let m' = shearing resistance in pounds per square inch:

$$m' = \frac{F}{9.55 \times (p + 2)}$$

at 2-in. penetration:

$$m' = \frac{22.67\ N^{1.01}}{9.55 \times 4} = 0.593\ N^{1.01}$$

at 3-in. penetration:

$$m' = \frac{28.33\ N^{1.01}}{9.55 \times 5} = 0.593\ N^{1.01}$$

at 4-in. penetration:

$$m' = \frac{34\ N^{1.01}}{9.55 \times 6} = 0.593\ N^{1.01}$$

$m' = 0.593\ N^{1.01}$ in pounds per square inch

$m' = 85.39\ N^{1.01}$ in pounds per square foot

CORRELATION OF PENETRATION MEASUREMENTS WITH OTHER SOIL RESISTANCE TESTS

Penetration Index and Shearing Resistance:

Among the laboratory tests the measurement of shearing resistance has been the most successful and appears to be the most significant. The shear test consists simply of placing the core of soil in a cylinder and pulling out a section of the core by application of transverse force. The test is illustrated in Fig. 6 which shows the shear cylinder and the method of loading.

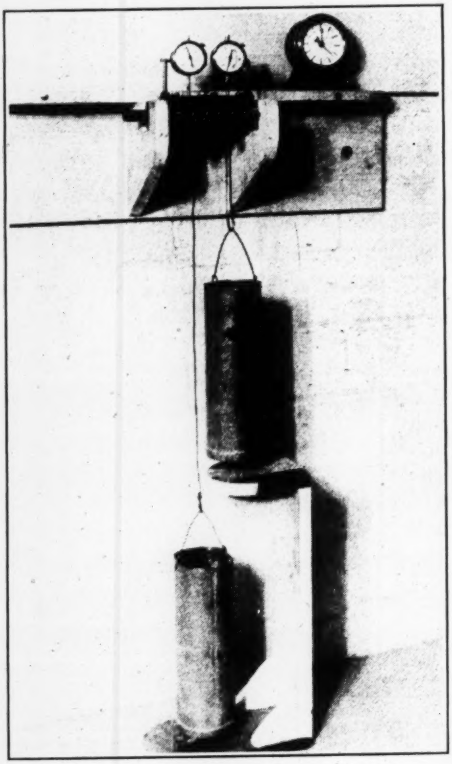


FIG. 6.—Shear Test Apparatus.

In general the procedure has been to measure the shear on two sections for each sample. The load is applied in increments which are held constant for a given period of time before another load increment is added in order to determine the load at which progressive movement occurs. The shearing displacement is measured accurately by means of deflection gages and the deformation for equal periods of time gives a measure of the rate of movement for loads in excess of the plastic limit. According to the fundamental law of plastic flow the rate of shearing displacement is directly proportional to the shearing stress in excess of the plastic limit. Thus a determination of this proportionality fixes the plastic limit or ultimate shearing resistance of the soil under static load.

In Fig. 7 the relation between shearing resistance m' and the penetration index N is shown with a final equation which represents a simplification which appears entirely satisfactory for practical application. The exponent of N in the equation for shearing resistance being only slightly greater than unity the equation may be approximated by a linear relation for the sake of simplicity. In such an approximation is made the best straight line is given by the following equation with the shearing resistance expressed in pounds per square foot:

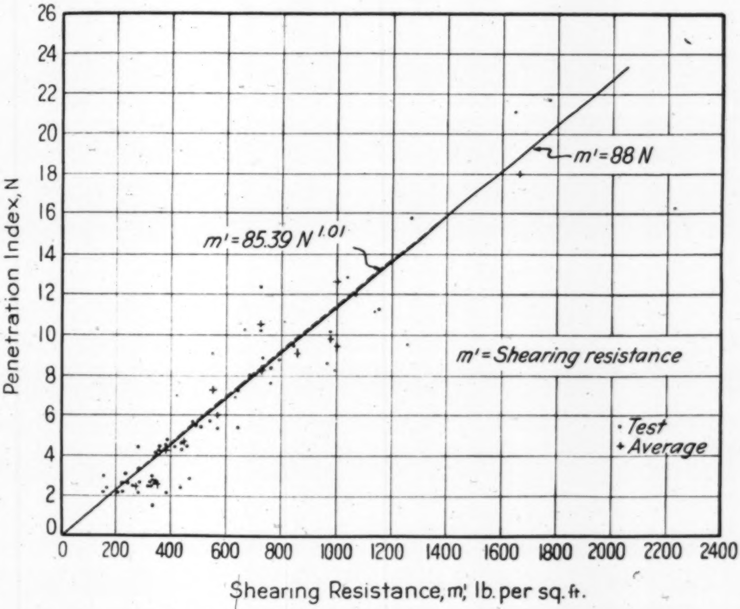
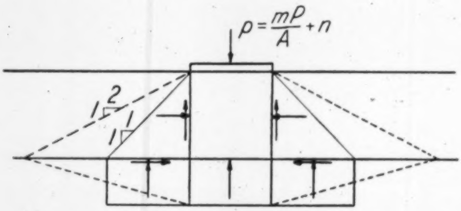


FIG. 7.—Correlation of Shearing Resistance and Penetration Index.

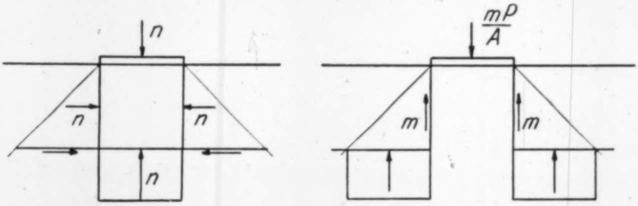
$$m' = 88\,N \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

The maximum departure of Eq. 2 from the original relation developed from the penetration data is only 1.5 per cent at a penetration index of 5 and 1 per cent at a penetration index of 50, these percentages representing the error at extreme values of the penetration index.

The results of laboratory shear tests are shown in Fig. 7 by plotting measured shear against penetration index N for individual samples as well as for the average of several samples at each elevation investigated. For perfect agreement between the laboratory shear test and the field penetration the plotted points should fall on the curve represented by equations developed from the penetration analysis. The values of shearing resistance as determined by penetration measurements and as measured in the laboratory show a good general agreement in spite of the dispersion of individual results which may be anticipated from the natural irregularities of soil structure. The results of investigation so far conducted make it quite clear that any method of measuring soil resistance must include sufficient duplication of tests to give a good average determination and eliminate the greater variation in individual samples. In fact one of the most difficult problems in soil testing is to obtain enough data to yield reliable results and still keep the multiplicity of tests within practical limits. In the data shown in Fig. 7 the practice has been to take from four to six samples at each elevation. While more samples would be very desirable, the investigations were in connection with actual construction projects and the information must be made available for design purposes without prolonging the field work and laboratory tests beyond an absolute minimum.



(a) Combined Stress Reactions



(b) Developed Pressure. (c) Perimeter Shear

FIG. 8.—Stress Reactions.

Penetration Index and Bearing Capacity:

The correlation in the case of bearing-capacity tests and penetration measurements is somewhat more involved inasmuch as the bearing-capacity determination itself requires a series of tests using different sizes of plates and the opportunities for such extensive tests are not nearly so frequent. Furthermore, the analysis of bearing-capacity tests, themselves, involve methods that are not entirely accepted. While there is not space in the present paper to cover this subject, it is necessary to outline briefly some of the conclusions drawn from bearing-capacity tests before a comparison with penetration measurements is possible.

In the author's opinion, investigation has shown that the ability of a body of soil to support surface loads can be described in terms of two types of resistance called stress reactions.[1] In Fig. 8 the conditions are shown and the stress reactions indicated in terms of which a straight-line equation for bearing capacity may be formulated:

$$p = \frac{mP}{A} + n$$

where p = bearing capacity in pounds per square foot,
m = perimeter shear in pounds per lineal foot,
P = perimeter in feet,
n = developed pressure in pounds per square foot, and
A = area in square feet.

In Fig. 8 (*a*) are shown the combined stress reaction in relation to the pressure distribution of the conventional type. In Fig. 8 (*b*) and (*c*) the separate stress reactions are shown to identify the stresses induced peculiar to each of the two types of resistance. The linear equation given has been found to represent the bearing capacity of different sizes of bearing area at any given amount of settlement for size ranges for which boundary conditions are similar. Available evidence indicates that one linear relation may be used for areas greater than about 1 sq. ft., and another such relation holds for areas less than approximately 16 sq. in. There is, however, a range between these sizes affected by changing boundary conditions which have not been thoroughly investigated. The variation in bearing capacity with the size of the area is controlled by the stress reactions arising from boundary conditions, and must be included in a complete conception of soil resistance.

It is true that perimeter shear does not furnish a large part of the total support of spread foundations or large mats but it is a very significant factor in test sizes of bearing areas. It is also subject to variation caused by changing the rigidity of the bearing area and several other conditions which must be carefully controlled in test procedure, but failure to accord it proper consideration makes any series of bearing tests quite misleading. When the boundary forces are properly treated the resisting pressure developed by a body of material may be accurately determined and becomes the controlling factor in ability to support loads. As indicated in Fig. 8 the developed pressure while definitely related to volume change or consolidation is dependent also on the confining influence of the surrounding body of soil. Thus the limiting value of the developed pressure or the bearing capacity limit is controlled by resistance to lateral displacement which depends in turn upon the shearing resistance furnished by the soil. If any loaded area stresses the body of soil in excess of its plastic limit, progressive settlement must follow due to lateral displacement. Very little if any of this progressive settlement can be charged to consolidation and consequently a decreasing rate of settlement and eventual equilibrium appears to be a vain hope.

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[1] Discussion by W. S. Housel of paper by C. A. Hogentogler and E. A. Willis on "Subgrade Soil Testing Methods," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 731 (1934).

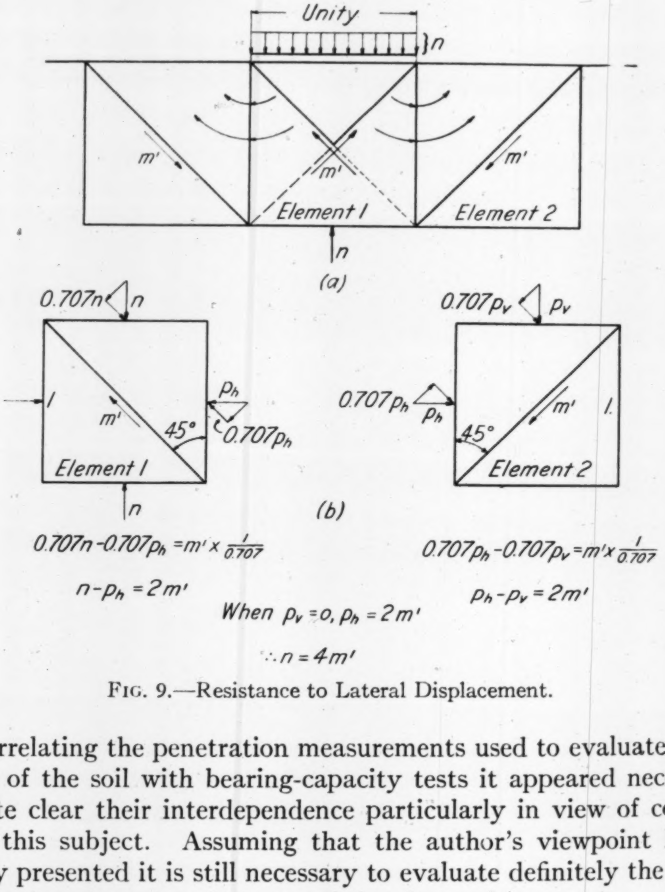
Unity
n
m'
m'
m'
Element 1
Element 2
n
(a)
0.707n
n
0.707p_v
p_v
1
p_h
m'
45°
0.707p_h
Element 1
n
0.707p_h
p_h
m'
1.
45°
Element 2
(b)
$0.707n - 0.707p_h = m' \times \frac{1}{0.707}$
$n - p_h = 2m'$
$0.707p_h - 0.707p_v = m' \times \frac{1}{0.707}$
$p_h - p_v = 2m'$
When $p_v = 0, p_h = 2m'$
$\therefore n = 4m'$

FIG. 9.—Resistance to Lateral Displacement.

In correlating the penetration measurements used to evaluate shearing resistance of the soil with bearing-capacity tests it appeared necessary to make quite clear their interdependence particularly in view of conflicting views on this subject. Assuming that the author's viewpoint has been sufficiently presented it is still necessary to evaluate definitely the ultimate limit of developed pressure in terms of shearing resistance.

In Fig. 9 is shown the manner in which the resistance to lateral displacement contributes to bearing capacity at the critical value of the developed pressure which determines the bearing-capacity limit. In Fig. 9 (*a*) is shown a bearing area of unit width and indefinite length subjected to a uniform pressure *n*. The discussion in this case is limited to the two-dimensional problem and considers only one stress reaction, developed pressure. In building up the general case the various stress reactions which enter into the bearing capacity may be superimposed, but in order to simplify the problem a separate treatment of each is preferred.

The pressure carried by the element directly beneath the bearing area depends primarily upon shearing resistance which furnishes the resistance to lateral displacement as well as making it possible to transmit some vertical load directly downward. For the case under discussion the horizontal and vertical planes which bound elements 1 and 2 in Fig. 9 are principal planes and the diagonal planes at angles of 45 deg. with the principal planes are planes of maximum shear. The conditions of equilibrium for these elements require that the difference between the greatest and least principal stress be less than twice the shearing resistance. As shown in Fig. 9 the critical value of the developed pressure occurs when the difference between the principal stresses is equal to twice the shearing resistance, at which point the developed pressure is equal to four times the shearing resistance.

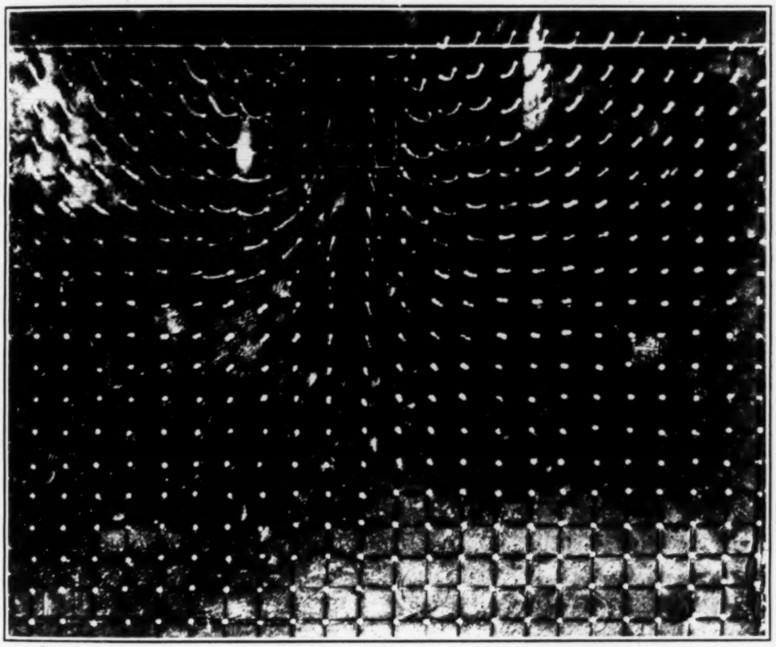

FIG. 10.—Showing the Movements of Plastic Clay Under a Bearing Area Loaded to Produce Progressive Settlement.

The emphasis placed on resistance to lateral displacement as the actual limit to bearing value of a body of soil and the relation between developed pressure and shearing resistance results from collecting and analyzing observations of soil behavior by a number of various methods. One type of study which has proved quite enlightening is shown in Fig. 10 illustrating the important connection between lateral displacement and bearing value. It is a photograph obtained by a time exposure of the movement of plastic clay under a bearing area loaded to produce progressive settlement. The exposed surface of the clay was marked off in a system of squares and white-headed nails were used to mark the corners of each square. The clay was confined by a glass plate which restricted the displacement to a lateral direction in accordance with the two-dimensional problem under consideration. The direction of the lines of displacement are clearly shown and agree essentially with the description given in connection with Fig. 9.

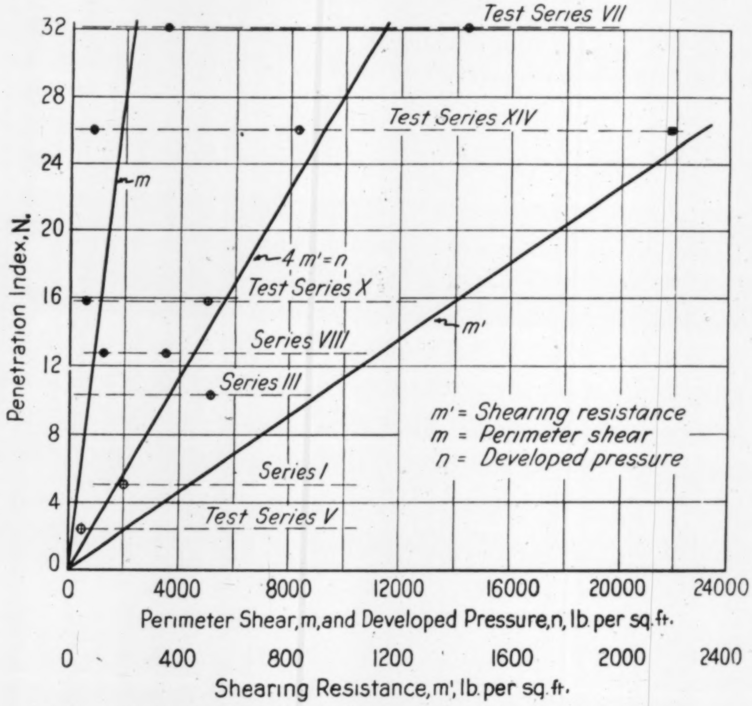

FIG. 11.—Correlation of Bearing Capacity and Penetration Index.

In Fig. 11 is shown the correlation of penetration index with the stress reactions which are the essential variables in bearing capacity. There are shown results from seven series of bearing-capacity tests for which penetration measurements were also obtained. As previously stated the developed pressure is the more important factor and the straight-line relation shown has been determined as four times the shearing resistance as measured by the penetration method. The values of developed pressure as determined by load tests are shown as the plotted points distributed about this line. The comparison indicates the general agreement of the two independent methods of measurement with a dispersion of individual determinations no greater than might be anticipated.

Another relationship shown indicates the values of perimeter shear for different values of the penetration index—in this case entirely empirical as there has not yet been found a rational method of correlating shearing resistance of soil with the boundary stresses. A line has also been shown indicating the values of shearing resistance in pounds per square foot, although only one independent test is shown in this case. In the series of tests conducted on the 26-blow soil, the opportunity was available to run a load test on a steel cylinder pile and it was found that the ultimate shearing resistance was 2180 lb. per sq. ft. as compared to 2290 lb. per sq. ft. from the penetration measurements.

In considering the comparison between soil resistance as determined by bearing-capacity tests and penetration measurements, the difficulty of obtaining representative samples by the latter method should be recognized as the major source of discrepancy in the results shown. The samples are taken by driving a comparatively small pipe into the soil at the elevation of the load tests and at several elevations below the test level. The average of the group of penetration measurements must represent the result of one method while the load tests themselves are the integrated results reflecting the variation in soil structure and irregularity in the zone affected by the load tests. Perhaps the average of a much greater number of samples would yield even better correlation but practical considerations again limit the extent to which results may be duplicated. Considering the idiosyncrasies of the material to be dealt with and the fact that it must be accepted as it is found with no opportunity whatsoever to control its properties the results seem reasonably satisfactory.

CONCLUSION

In conclusion of the presentation of the penetration method of measuring soil resistance it is pertinent to point out the opportunities for improvement and probable development in the future. The results up to the present time indicate that the method is a very promising one and sufficiently flexible and inexpensive to increase greatly the projects for which more definite measurement of soil resistance may be made available. The method has been used in a number of projects on which it would not have been possible to run the more expensive load tests and the results have been regarded with considerable favor. It appears that there should be more opportunities for complete correlation of results before final acceptance of the penetration measurements as a standard method is justified and there is particular need for amplification of the data for very soft and very stiff soils. In the discussion it was indicated that the equations for penetration per blow were subject to revision and this is especially true at both extremes of the range for which these relations were determined. Considerable difficulty has been encountered in correlating results for very soft and very stiff soils and while improvements in test procedure both in the field and laboratory are being made, more fundamental changes may be required.

When the method has been given sufficient study and its reliability demonstrated and generally accepted there are certain mechanical improvements that would be desirable. A smaller and more compact machine which would be operated in a small casing has been considered and seems entirely feasible. A more satisfactory method of recording penetration measurements has been studied and may also be a desirable improvement. The problem of sampling under water has presented itself and is one that requires special design but otherwise presents no insurmountable obstacles.

STABILITY OF GRANULAR MIXTURES

By D. S. Berry[1]

Synopsis

The development of a practical method for measuring the stability of mixtures for use in flexible type road construction is the primary object of the general investigation being carried on in the Michigan State Highway Laboratory at the University of Michigan. The first project has been carried out for the purpose of developing adequate testing equipment and to investigate the factors affecting the stability of granular materials.

A machine has been designed for making actual bearing capacity tests on prepared samples to determine load-carrying capacity and deformation under load. Shearing-resistance apparatus has been constructed to investigate the possibility of using a simplified shear test as a measure of stability. A vibration method has been developed for controlling the degree of compaction of the materials. The practicability of this equipment for use in the design of surface mixtures is discussed and plans for future research are described.

The results of the first tests are presented as supporting evidence for the following conclusions, applicable to granular materials.

1. Internal resistance or stability depends primarily on mechanical arrangement or interlocking of the individual particles.
2. Stability may be measured in terms of bearing capacity or shearing resistance.
3. Bearing capacity is related to boundary conditions as reflected in size of loaded area and the depth of the material.
4. Shearing resistance is a function of normal pressure, but does not appear to be adequately defined by existing conceptions of internal friction.

Introduction

This investigation has been made as a part of a general study being carried on by the Michigan State Highway Department for the purpose of developing a rational method for the design of surface and base courses for flexible type low-cost roads. Since such roads must carry loads without serious displacement, the internal stability of the materials and mixtures becomes an essential property governing their use. In the surface course it is needed to prevent failures due to shoving or rutting, and in base courses it is necessary to distribute the load properly over the subgrade. The required thickness of these two component parts is determined by stability, properly correlated with the stresses resulting from the application of surface loads.

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[1] Fellow in Civil Engineering, University of Michigan; and Research Assistant, Research and Testing Division, Michigan State Highway Dept., Ann Arbor, Mich.

It is apparent, therefore, that a measure of this stability is very necessary in order that the design may be placed on a rational basis. The use of a direct loading test in the laboratory, measuring the bearing capacity of different sizes of test areas will approach the actual field conditions. Such a test may be made on the actual material mixtures of thickness corresponding to the actual pavement thickness, and the results should give a practical measure of stability.

This first project has been carried out with three purposes in mind, (1) to develop adequate apparatus for making bearing-capacity tests on surface mixtures, (2) to investigate the possibility of using simplified shear tests as a measure for stability, and (3) to identify and measure some of the fundamental elements of internal stability of granular materials.

Considerable time has been spent in the design and development of accurate testing equipment and the establishment of test technique. The first instrument to be constructed was a loading machine for making bearing tests on prepared samples to determine load-carrying capacity and deformation under load. A study of compaction by mechanical vibration has been made and a method devised to obtain accurate control of the density of the materials. An instrument for measuring shearing resistance has been designed that gives accurate and consistent results for fine materials.

The first actual testing work has been confined to a study of the stability of dry granular materials. The actual factors affecting their load-carrying ability being investigated are:

1. The density or degree of compaction of the material.
2. The size of the bearing area.
3. The thickness of the sample of material beneath the area of load application.
4. The type of material.
5. The relation between shearing resistance and bearing capacity.

THEORY

A granular material is a heterogeneous mass of individual particles, and as such cannot be classified as an elastic medium. The ultimate stability of such a material and the deformations that occur when the material is loaded depend not only upon the characteristics of the individual particles but also upon the state in which the individual particles exist and upon dimensional effects that result from particular conditions of loading. For these reasons, it is very difficult, and up to the present time impossible, to set up an exact mathematical theory of stability. Under such conditions testing procedure must simulate actual conditions encountered in practice and measure directly those properties of fundamental importance.

Bearing-Capacity Tests:

The measurement of the resistance of a material to direct surface load is one of the most satisfactory means of measuring stability. This method approximates conditions existing in the application of wheel loads on actual road surfaces and permits a determination of the effects of size, shape and rigidity of the loaded areas, as well as the thickness of the material sample. The results of such tests may later be used to correlate the results of simple shear tests with actual load-carrying capacities of road surface mixtures.

The movement of granular material beneath a bearing plate as observed in actual tests is illustrated in Fig. 1. The wedge of material directly below the plate is confined and moves downward with the bearing area. The material outside this wedge moves downward, outward and upward as the ultimate shearing resistance of the material is exceeded along surfaces of maximum shear or minimum resistance to shear.

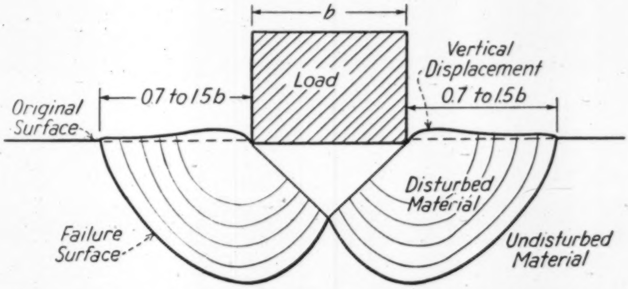

FIG. 1.—Diagram Showing Failure of Granular Material Beneath a Bearing Plate.

The settlement under the bearing plate may be divided into two parts: (1) settlement due to consolidation and elastic deformation, and (2) settlement due to lateral displacement. The resistance to consolidation is limited only by the crushing strength of the particles while resistance to lateral movement is limited by the shearing strength of the material along the failure surfaces shown. The ultimate bearing capacity is reached just before this resistance to lateral displacement is exceeded.

Shear Tests:

Actual measurements of the shearing resistance of a material, made at different intensities of normal pressure, determine the relation between tangential and normal components of stress acting on any plane at ultimate values coincident with failure. The ratio of these two stresses has been expressed by Rankine and Coulomb as the tangent of the so-called "angle of internal friction."

The determination of shearing resistance is important, not only because it governs the ultimate load-carrying capacity of the material, but also because it is the factor that controls the rate of distribution of the load through the layer of material. The shearing resistance of the material on planes passing through the boundary of a loaded area is a measure of that portion of the load distributed into material surrounding the loaded area. Shearing resistance thus becomes a primary factor in the design of base courses of proper thickness to distribute the load over the subgrade.

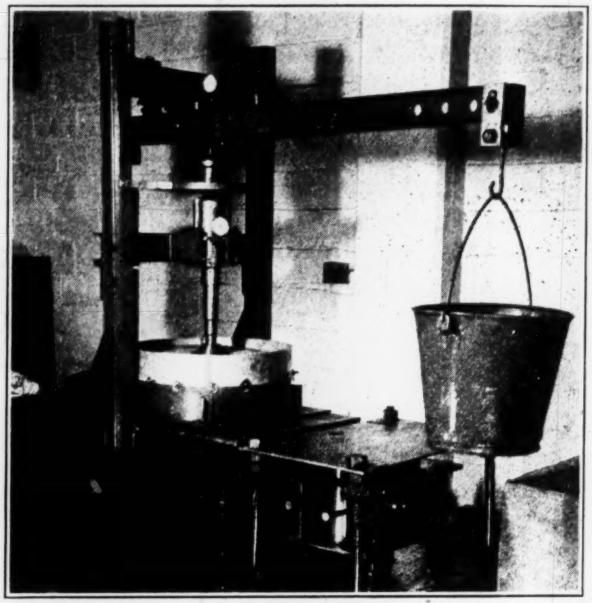

FIG. 2.—Apparatus for Measuring Bearing Capacity.

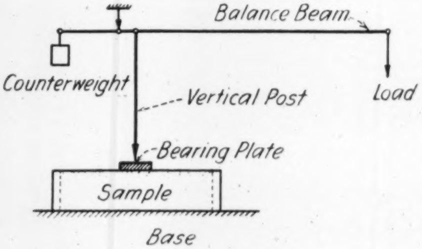

FIG. 3.—Diagram of Bearing Capacity Machine.

TEST APPARATUS

Bearing-Capacity Machine:

The apparatus used for the bearing-capacity tests is shown in Fig. 2. The loading machine consists essentially of a balance beam suspended from a heavy steel framework. Figure 3 shows the forces acting in the machine. The vertical loading post is provided with an accurate vernier screw used to keep the balance beam level as the material deforms under the bearing plate. Ball bearings are provided between all moving parts in order to reduce friction to a minimum. Precision methods used in the construction of the apparatus make it possible to measure the settlement of the bearing plate to 0.001 in. throughout the entire load range of 5000 lb.

This machine is used for measuring bearing capacities on samples 19 in. in diameter and up to 10 in. in depth, with different sizes of bearing plates up to 32 sq. in. in area. Tests may be run not only upon samples used in surface courses and base courses, but also upon combinations of the two surfaces.

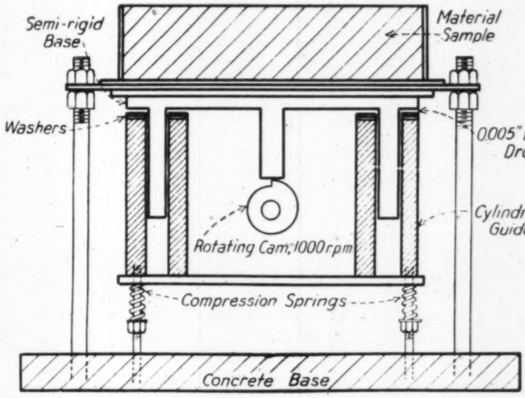

FIG. 4.—Schematic Diagram of Compaction Vibration Machine.

Compaction Vibration Machine:

The mechanical compaction vibrator, a modification of the design of the machine used by the Bureau of Public Roads for vibrating concrete beams,[1] is shown in Fig. 2. A schematic diagram, Fig. 4, shows the essential working parts. The semi-rigid table is held stationary by means of heavy vertical bars. The rotating cam and the compession springs keep the inner part of the machine oscillating back and forth, striking the under side of the table twice for each revolution of the cam. The degree of compaction of the material may be changed by varying either the frequency, intensity or time of vibration.

This vibration machine will compact granular material to densities much greater than may be obtained by hand tamping. Although it has not been used as yet for the compaction of bituminous mixtures, it is anticipated that it can be used with slight modification for compacting such mixtures to the density of actual road surfaces.

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[1] W. A. Blanchette, "Some New Relations Bearing on Concrete Mixtures," *Public Roads*, Vol. 15, May, 1934, p. 58.

Shearing Equipment:

The shear test apparatus is shown in Fig. 5. It consists essentially of a special shearing cylinder for holding the material sample, two balance beams for applying normal load to the ends of the sample, and a cable-pulley arrangement for applying transverse force to a section of the cylinder. A diagram showing the forces acting is shown in Fig. 6 (*a*).

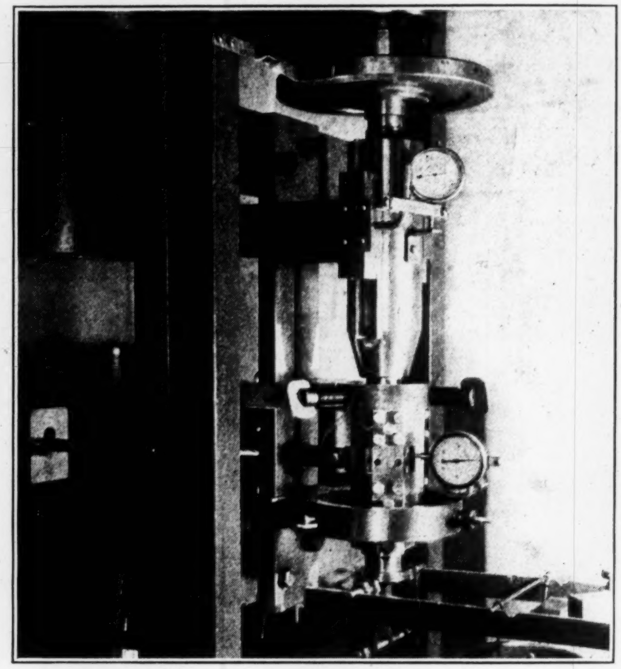
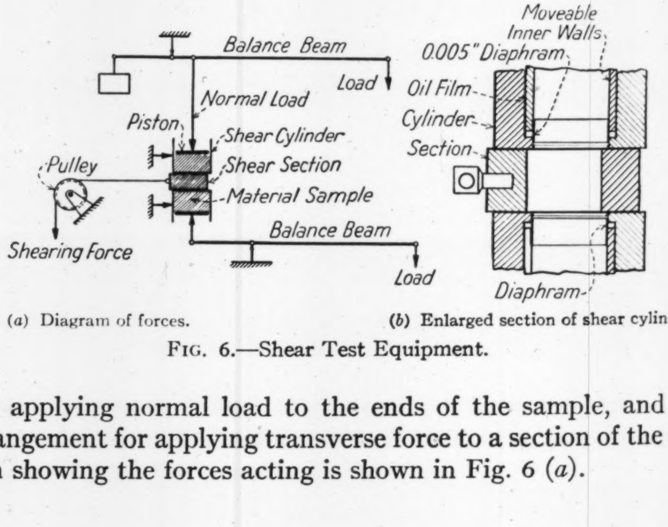


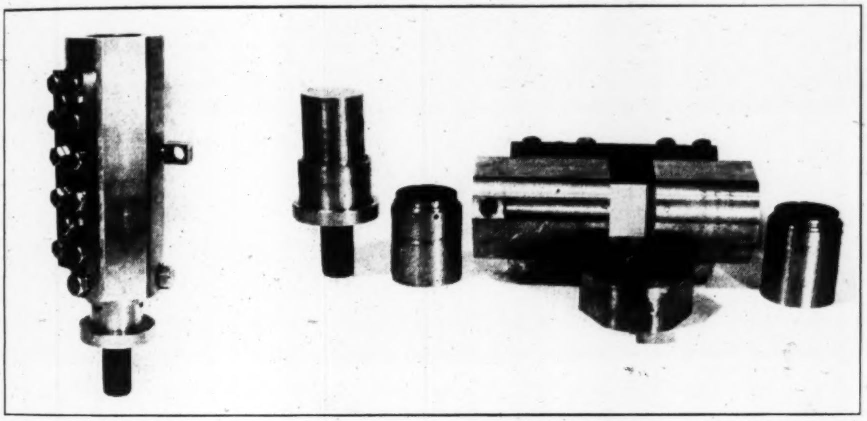
FIG. 5.—Shear Test Equipment.


Balance Beam
Load
Normal Load
Piston
Shear Cylinder
Pulley
Shear Section
Material Sample
Balance Beam
Shearing Force
Load
Moveable Inner Walls
0.005" Diaphram
Oil Film
Cylinder
Section
Diaphram

(*a*) Diagram of forces. (*b*) Enlarged section of shear cylinder.

FIG. 6.—Shear Test Equipment.

Preliminary tests[1] similar to the tests reported by Bell in 1914[2] were made with a simple shear cylinder having a rigid base rather than a movable lower piston as shown in Fig. 6. These preliminary tests brought out two sources of discrepancy which necessitated changes in the design of the apparatus. It was found that when shearing failure took place there was a considerable volume expansion as the particles on the shearing surface rolled over one another. This phenomenon has been noted by Terzaghi.[3] In the first place the sample rested on a rigid base and all of the volume displacement took place in an upward direction, disturbing the mechanical arrangement of the particles at the shear section. In the second place frictional resistance along the walls of the shear cylinder apparently caused considerable variation in the intensity of normal components at the shear section which seriously affected the results.



(*a*) Assembled. (*b*) Unassembled.

FIG. 7.—Views of Shear Cylinder.

The double balance beam with movable pistons top and bottom and the shear cylinder with movable inner walls were adopted in order to permit volume expansion or consolidation of the material to take place and at the same time to maintain constant normal pressure at the shear section. The detail of the modified shear cylinder is shown in Fig. 6 (*b*) and photographs of this apparatus are shown in Fig. 7.

This instrument permits the direct measurement of the shearing resistance in a simple and easy manner. Although the small diameter of the test cylinder limits it to the testing of fine materials, it is possible that a larger size of cylinder could be used for making a simple routine test for the stability of actual road surface mixtures.

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[1] Discussion by W. S. Housel of paper by C. A. Hogentogler and E. A. Willis on "Subgrade Soil Testing Methods," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 729 (1934).

[2] A. L. Bell, "The Lateral Pressure and Resistance of Clay," *Proceedings*, Inst. Civil Engrs. (London), Vol. CXCIX, p. 243 (1914–1915).

[3] Charles Terzaghi, "Old Earth Pressure Theories and New Test Results," *Engineering News-Record*, Vol. 85, September 20, 1920, p. 632.

TEST PROCEDURE

Bearing-Capacity Tests:

A definite weight of dry aggregate is placed in the mold and the material is vibrated on the compaction vibrator until the predetermined compacted depth of the material has been attained. A flat steel plate is placed on the surface of the material during the vibration period in order to keep the upper surface level and to facilitate the accurate measurement of depth. This method permits one to control the density of the aggregate accurately.

This sample is then placed under the loading post of the bearing-capacity machine, the bearing plate is placed on the surface of the material, the balance beam is leveled with the loading post in contact with the bearing plate, the deflection dial is set at zero, and the sample is ready for testing. Load is applied in increments at constant time intervals. Deflections may be read either from the gage of the screw vernier or from an Ames dial.

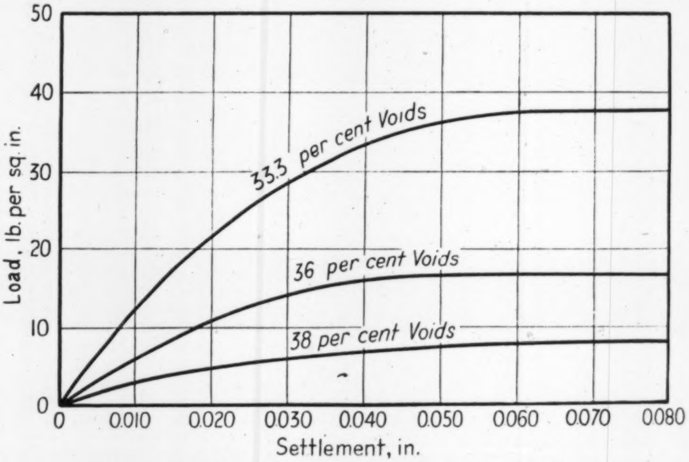

FIG. 8.—Effect of Density of Material on Stability.
Bearing capacity tests on standard Ottawa sand. 6.5-in. depth of sand. 32 sq. in. area of round bearing plate.

The loading is continued until failure of the sample occurs. This failure is accompanied by lateral and upward movement of the material around the loading area, similar to the rutting of an actual pavement surface course.

Shearing-Resistance Tests:

A definite weight of aggregate is placed in the shear cylinder and it is vibrated with a light plunger on the surface of the material until the compacted depth has been reached. The shear cylinder is then clamped in position in the loading machine, and the desired normal loads are applied to the top and bottom of the cylinder. The restraining bolts are removed from the bottom plunger, shear section and inner walls, and the sample is ready to be tested.

Transverse load is applied to the shear section in increments. Horizontal and vertical deflections are read for each load increment. The load is increased until the material has developed its ultimate shearing resistance after which sudden failure occurs.

MATERIALS

Standard Ottawa sand has been used for many of the tests in order to eliminate some of the variables that exist in granular materials. Some tests have been made on different gradings of a sharp concrete sand of No. 8 maximum size. Tests have also been made on different sizes of chilled lead shot.

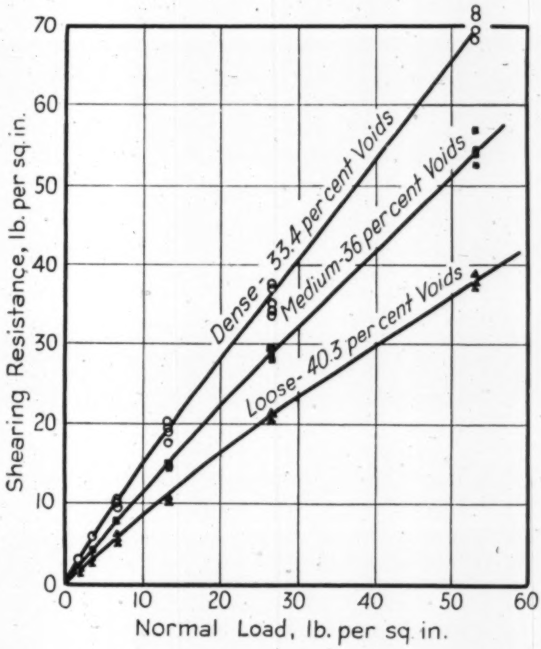

FIG. 9.—Effect of Density on Shearing Resistance.
Standard Ottawa sand, vibrated to compaction desired.

PRESENTATION OF TEST DATA

Effect of Degree of Compaction:

The load-settlement curves in Fig. 8 show that the degree of compaction of a granular material is one of the most important factors affecting bearing capacity and settlement under load. Each curve at degrees of compaction of 38, 36 and 33.3 per cent of voids represents the average of 3 to 6 load tests run at that percentage of voids. Although Ottawa sand

Geo. L. Smith

poured loose has from 40 to 42 per cent of voids, 38 per cent was the maximum which could be obtained with any uniformity. The value of the ultimate bearing capacity at a maximum density of 33.3 per cent of voids is several times the value at a density of 38 per cent of voids. Settlements that occur for a given unit load are proportionally greater as the density of the material decreases. Similar data obtained with different sizes of plates and on different thicknesses of material show substantially the same relations.

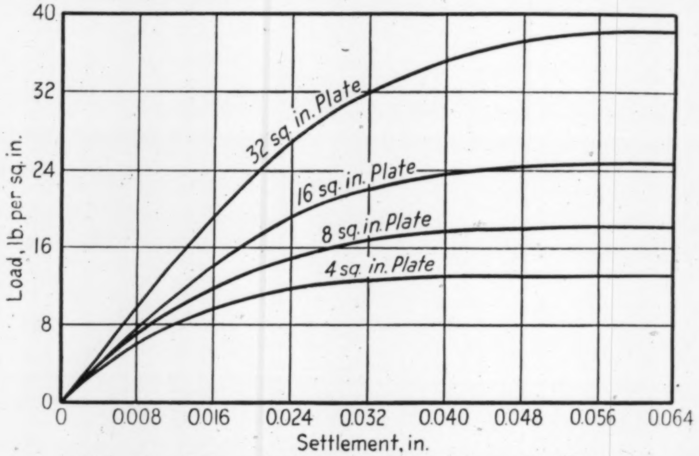

FIG. 10.—Effect of Size of Bearing Plate on Stability.
Bearing capacity tests on standard Ottawa sand. 33.4 per cent of voids. 6.5 in. depth of sand.

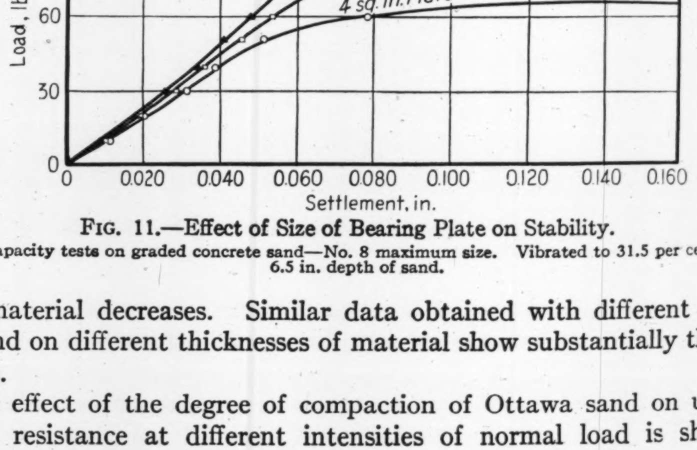

FIG. 11.—Effect of Size of Bearing Plate on Stability.
Bearing capacity tests on graded concrete sand—No. 8 maximum size. Vibrated to 31.5 per cent voids. 6.5 in. depth of sand.

The effect of the degree of compaction of Ottawa sand on ultimate shearing resistance at different intensities of normal load is shown in

Fig. 9, each point representing a separate shearing test. At a given intensity of normal load there is a marked increase in shearing resistance as the percentage of voids decrease, indicating the fundamental importance of mechanical arrangement or interlocking of particles as a primary source of internal resistance. The slope of the curves in Fig. 9 becomes practically constant after an initial stage in which there is evidence of some slight shearing resistance which is not directly proportional to normal pressure.

Effect of Size of Bearing Plate:

Figure 10 shows the effect of the size of test plate on bearing capacity for standard Ottawa sand. Each curve represents the average of several test series. The load-settlement diagrams in Fig. 11 show the effect of size of area for a graded concrete sand with a No. 8 maximum size. It is apparent for the size range investigated that the bearing capacity at a constant settlement is greater for the larger sizes of bearing plates. Although in the lower range of bearing capacity this variation is not so marked, the effect of the size of area on the ultimate load-carrying capacity is much greater. While these results are not in agreement with certain statements that the bearing capacity of granular materials is but slightly affected by the size of the bearing area, they are in agreement with the results of extensive investigations of Scheidig,[1] and Kogler.[2]

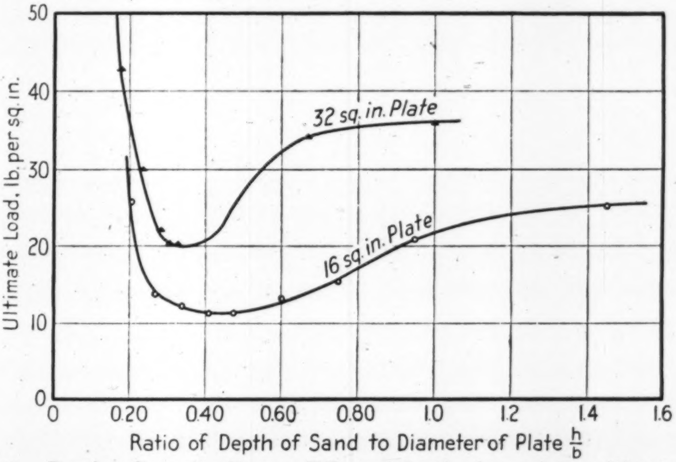

FIG. 12.—Bearing Capacity Tests. Effect of depth of sample on ultimate load. Standard Ottawa sand. Maximum compaction. 33.4 per cent voids.

Effect of Depth of Material:

The curves in Fig. 12 show the variation in ultimate load-carrying capacity of Ottawa sand with the ratio of depth to width of bearing area.

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[1] A. Scheidig, "Neure Verfahren in der Analyse und Vorhersage von Bauwerksetzungen," *Bautechnik*, Vol. 11, No. 12, p. 15 (1933).

[2] F. Kogler, Discussion on "Soil Mechanics Research," *Transactions*, Am. Soc. Civil Engrs., Vol. 98, p. 299 (1933).

It will be noted that the effect of depth must be considered with the size of the loaded area in order to be significant. Each point represents the average value of two or more separate tests. An important factor affecting this group of tests was the lack of horizontal friction between the bottom of the sample of material and smooth iron plate on which it rested. The resistance to lateral displacement was substantially decreased at depths at which the plane of maximum horizontal shearing stress approached the plane of the base, but becomes very high for shallow depths where direct arching action controls.

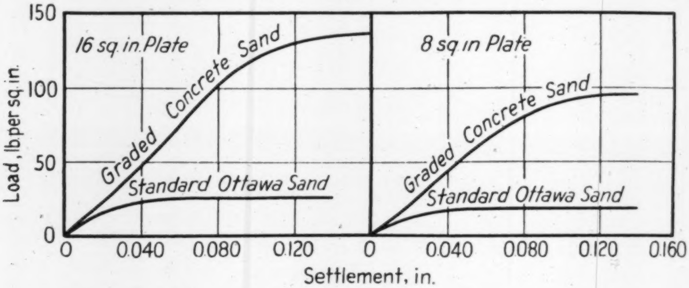

FIG. 13.—Effect of Type of Material on Bearing Capacity.
Vibrated to maximum compaction. 6.5-in. depth.

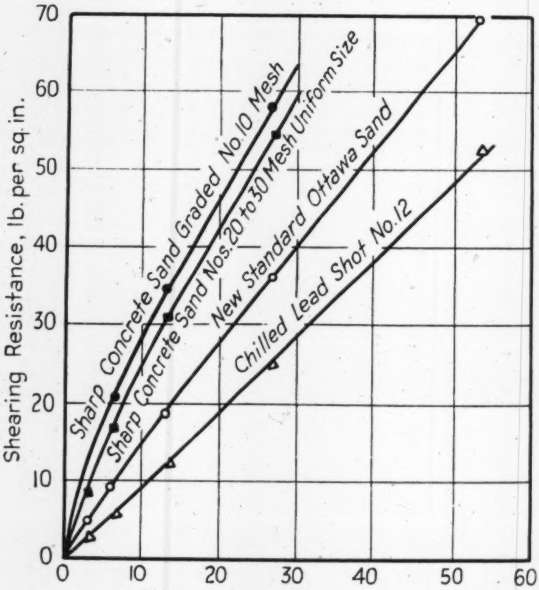

FIG. 14.—Effect of Type of Particle on Shearing Resistance.
Maximum compaction by vibration.

Later, duplicate tests were made with a heavy sand-glue bond coat on the iron base plate. The results of these tests showed that there was no apparent decrease in ultimate load as the depth of material was decreased.

Effect of Type of Material:

The data in Fig. 13, transposed from Figs. 10 and 11, may be used to compare the relative bearing capacities of Ottawa sand and graded concrete sand. It should be noted that the ultimate load-carrying capacity of the concrete sand is in each case several times that of the Ottawa sand.

Figure 14 shows the effect of the type of material on the ultimate shearing resistance at different normal loads. The materials were tested at maximum compaction and consisted of chilled lead shot, standard Ottawa sand, concrete sand of uniform size and the same concrete sand graded finer than No. 10. The lead shot is spherical, the Ottawa sand is only approximately spherical, while the concrete sand is relatively sharp and angular. While the shearing resistance curve for the uniform size concrete sand lies only slightly below that for the graded concrete sand, it is in turn much higher than the shearing resistance curves for the other types of uniform size material, indicating that particle shape is probably more important than gradation in the stability of granular material.

The data in Fig. 14 show clearly the marked increase in shearing resistance for angular materials and verifies the previous conclusion that mechanical arrangement or interlocking is one of the most if not the most important source of internal resistance in granular materials.

DISCUSSION OF DATA

Bearing-Capacity Tests:

The variation in bearing capacity with the size of bearing area at any given settlement as shown in Figs. 10 and 11 is one of the most important factors in the stability of granular masses. It is apparent from the conditions of the tests and the nature of this variation that it is directly associated with dimensional effects which are dependent upon boundary conditions. If there were no such variation, all sizes of area should support equal intensities of load at a given settlement. It becomes necessary, therefore, to consider the various conditions to which individual elements are subjected at the boundary of the loaded area as compared to points removed from the boundary and to study the manner in which the material reacts under each situation.

When an element under the center of the plate is considered it is apparent that there is sufficient confining influence from the surrounding elements to enable it to develop some resistance to lateral displacement. However, an element under the edge of the bearing plate at the surface has only a limited lateral resistance available from the surrounding material, and it becomes a source of weakness. This results in a deficiency in supporting capacity at the perimeter of the plate.

The ability of the material to support load, therefore, depends upon elements beneath the central portion of the loaded area which have sufficient lateral support to develop resistance to displacement under vertical load.

As the size of the plate increases, the boundary weakness becomes relatively less effective: a variation which may be expressed as a function of the ratio of the perimeter to the area (the P/A ratio as used by W. S. Housel in the investigation of settlement in clay).[1] The actual data are in accordance with this analysis, since it was found that bearing capacities of the larger areas are greater.

When a cementing medium is added to the granular material, as in the case of mixtures used in flexible type road construction, an additional factor is introduced. The cohesion of this cementing medium introduces a definite resistance to shear on the perimeter of the plate at the surface of the mixture. Since cohesive forces require little deformation to develop them, it appears that this perimeter shear resistance will be present before the consolidation deformation of the central column of material has developed the resistance of this central column. The perimeter in this case would be a source of strength rather than weakness. For clays possessing only cohesion, research on foundations has shown that the perimeter is a source of strength for the entire range of loads and that the bearing capacities under small plates are greater than for the large plates.[1]

It is probable, therefore, that the properties of mixtures used in flexible type road surfaces will lie somewhere between the two extremes of a granular material and a clay, the relative position depending upon the type and amount of cementing medium used. Further research is planned to evaluate these properties for oil-aggregate mixtures and to develop test procedure for measuring the stability of such mixtures.

The variation in bearing capacity with degree of compaction has already been pointed out in connection with Fig. 8. A study of these data shows the fundamental importance of mechanical arrangement of particles on stability, since the variation in percentage of voids is directly associated with the degree of interlocking obtained. The importance of mechanical arrangement as affected by particle shape and grading is demonstrated by the data in Fig. 14, comparing the bearing capacity of graded concrete sand and standard Ottawa sand at maximum compaction. These data show that the graded concrete sand with sharp, angular particles has developed several times as much supporting capacity as Ottawa sand with only slightly angular particles. Similar tests conducted on chilled lead shot showed almost negligible supporting capacity.

The data showing the effect of depth of sample are very interesting from the highway standpoint. The tests made when the sample rested on the smooth iron base showed that the ultimate bearing capacities decreased as the depth was decreased to a certain point, because of lack of bond between the material and the base. As the depth of the sample was decreased still farther, the direct arching action through to the base carried a greater share of the load and the bearing capacities increased to very high values. When tests were made with a sand-glue bond coat between the base and the sample there was no deficiency in lateral resistance with a decrease in depth and the ultimate bearing capacities remained approximately the same until affected by the direct arching action. This study indicates the value of good bond between base course and relatively thin surface courses.

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[1] W. S. Housel, "The Bearing Power of Clay Is Determinable," *Engineering News-Record*, Vol. 110, No. 8, February 23, 1933, p. 244.

Shear Tests:

The significance of the shearing resistance tests have been pointed out in the presentation of the data. The important influence of mechanical arrangement and particle shape on shearing resistance which is apparent from these data indicates that some revision of the conventional conception of friction as a source of internal resistance is necessary. It is not clear how the following observations can be described by existing conceptions:

1. In most cases for compacted materials the ratio of tangential components to normal components of stress, generally referred to as the tangent of the angle of internal friction, is greater than unity, resulting in so-called angles of friction as high as 70 deg. The minimum value for this angle, obtained for chilled lead shot, was 43 deg.

2. There is also noted large variations in this angle with changes in degree of compaction.

3. As transverse shearing force is applied, there is motion normal to the shearing plane as well as tangential movement, indicating a stage of volume expansion previous to failure which introduces static reactions between individual particles not accounted for in existing theory.

4. There is a variation in the so-called coefficient of internal friction, noted in the lower range of normal load in the various tests as a static resistance due to interlocking, which is not directly proportional to normal pressure applied.

For materials without cohesion, the shearing resistance may conceivably be separated into two parts, (1) the resistance resulting from the static reactions of the particles, proportional to normal load, and (2) a factor of resistance due to the interlocking of the individual particles, which may vary only to a limited extent with normal load. For example, tests on chilled lead shot show a ratio of shear stress to normal stress that is practically constant for the entire range tested. On the other hand the tests on sharp concrete sand show much larger tangential components for any given normal pressure with considerable interlocking effect in the initial stage which is not directly proportional to normal load.

Materials with cohesion exhibit a definite shearing resistance at zero normal load, as shown by tests on clay.[1] It is evident, therefore, that shear tests of mixtures for flexible road surfaces will show both cohesion and interlocking, the relative proportion depending upon the amount of binding material present. No tests have been run on such mixtures as yet.

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[1] W. S. Housel, "A Penetration Method of Measuring Soil Resistance," see p. 472.

Relation Between Bearing Capacity and Shear:

The marked increase in both bearing capacity and shearing resistance with comparatively small increases in degree of compaction or changes in particle shape indicates that bearing capacity and shearing resistance are definitely related. This relation is difficult to establish mathematically as it involves such complications as a non-uniform distribution of pressure on the loaded area as well as varying intensities of normal pressure within the granular mass, which accompany the lateral distribution of vertical load. There must also be considered the dimensional effects which are the result of variations in size of loaded area and depths of material. The present status of this problem indicates that the possibility of establishing a definite mathematical relationship is promising, although such an important step should not be taken without further supporting evidence.

Proposed Development:

Apparatus has been developed for measuring bearing capacity and shearing resistance of material mixtures. This equipment has been used to measure some of the factors affecting bearing capacity and shearing resistance of granular materials. Further equipment to be used with the existing apparatus in the development of a laboratory design test for road surface mixtures is in process of construction.

The bearing-capacity test is believed to be an excellent research tool for measuring the stability of road materials under conditions simulating the action of surfaces under wheel loads. It may possibly be developed into a design test for surface mixtures, or be used with a simpler type of test for correlation with actual bearing capacity.

The present shearing-resistance test gives good results for fine granular materials. It is simple, rapid, and not affected by the personal equation. Some further study is necessary to determine to what extent the results of the shear tests may be correlated with actual bearing capacities. The present size of shear cylinder, however, cannot be used with materials containing coarse aggregate, and a larger shear cylinder may be necessary to eliminate this objection.

Equipment for measuring the shearing resistance of mixtures, combining the good features of both the above types of tests is now in the process of development. In this modified test it is proposed to evaluate stability by measuring the difference between the greatest and least principal stress a material will maintain before failure takes place by lateral displacement. Such a test would be a direct measure of the shearing resistance developed under conditions more nearly approximating the actual bearing-capacity tests and at the same time eliminate some boundary effects which complicate the analysis. This equipment is intended for use as a design test for surface mixtures of the oil-aggregate type.

CONCLUSIONS

While the results of the investigation to date have largely been used to point the way for future research, the following conclusions may be drawn from the analysis of present data:

1. Internal resistance or stability of granular materials depends primarily on mechanical arrangement or interlocking of the individual particles of the mass which is greatly affected by the degree of compaction and particle shape or angularity and to some extent by gradation.
2. Stability may be measured in terms of shearing resistance or bearing capacity under surface load and these fundamental properties are related.
3. Shearing resistance of a granular material is a function of normal pressure, but the relation between normal and tangential components of stress does not appear to be adequately defined by existing conceptions of internal friction.
4. Bearing capacity under surface load varies with the size of the loaded area and is controlled by boundary conditions which may be expressed in terms of the dimensions of the bearing area.
5. Resistance of granular materials to displacement under surface load is materially decreased by a deficiency in shearing resistance on horizontal planes at depths less than the width of the loaded area.

Acknowledgment:

The opportunity to conduct the investigation reported herein was made possible by Fellowships granted by the University of Michigan through the Civil Engineering Department. These grants included the Detroit Edison Fellowship, 1933–1934, and a University Fellowship for the year 1934–1935. The scope of the work has been greatly increased through the cooperation and financial assistance of the Michigan State Highway Department.

The author wishes to express appreciation to the following: Professor W. S. Housel, Research Consultant, Michigan State Highway Department, under whose direct supervision and guidance the work has been done; and John W. Kushing, Research and Testing Engineer, Michigan State Highway Department, whose cooperation has permitted the project to be carried out.

DISCUSSION

MR. I. F. MORRISON[1] (*by letter*).—The real value of this paper lies in the fact that it tends to clarify and develop some of the fundamental concepts in soil mechanics which have been gradually taking form in the engineering mind during the last decade. Until clear conceptions of such fundamental notions, as, for example, what Jacob Feld has called the "coefficient of internal resistance" and many other phenomena, are fully developed, further progress in this most important subject will be retarded. This paper exhibits the clear thinking which the subject demands and stands out in contrast to much of that unsound thought with which the topic is still befogged.

It is perhaps pertinent in a discussion of this sort to indicate alternative points of view with the hope that such remarks will not be mistaken for destructive criticism. The author states, under the theoretical considerations, that a granular material cannot be classified as an elastic medium. The elastic state, as such, is a property of all media and it is only when subjected to sufficiently high shearing stresses that it passes over into the plastic state.

With the rapid advance in knowledge concerning all kinds of materials, the classification of materials into elastic and non-elastic or plastic is being given up as inappropriate. It is true that sand, for instance, does not possess the characteristics usually associated with the concept plastic. Nevertheless, when it is deformed *en masse*, the process by which the deformation takes place is closely akin to the process of plastic deformation of all other materials and especially so as regards the requisite state of stress. Plastic deformation, however, is usually considered to take place without change of volume, whereas there is usually a change in volume for granular materials.

In the accompanying Fig. 2, one of Kögler's curves has been reproduced. The writer has added two curves with dotted lines to show that this type of curve may arise as a result of two independent terms, which appears to indicate that one should look for two primary influences. The writer does not care to suggest at present what these may be. Some work along mathematical lines has been done on this question by Fröhlich; but much, especially of an experimental nature, still remains to be done concerning this anomaly. The explanation does not appear to be dependent on the absence of cohesion on the one hand and its presence on the other. Kögler's curves are for cohesionless sand so that cohesion is not a necessary condition.

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[1] Professor of Applied Mechanics, Department of Civil and Municipal Engineering, University of Alberta, Edmonton, Alta., Canada.

There appears to be, in a granular material below a loaded bearing plate, three regions defined by the state of stress which exists in them. These are best illustrated by a sketch, as shown in the accompanying Fig. 1 following Kögler and Scheidig. The regions have been indicated as I, II and III. In region I the material is said to be in the disturbed state. It has been slightly compressed vertically and, being granular in character, has probably undergone some volumetric change due to distortion by shearing. Anyone who observes what happens when he places his foot on hard moist sand will see the effect of a volumetric expansion—the water from region III passes into region I. In region II the material is under stress but is in the elastic state and acts as a support for region I. In region III, provided the mass cannot sustain tensile stress, there is, in so far as the surface load is concerned, no stress. To all of these stresses the effect of the body forces should be added. These effects are not appreciable, however, in the case under discussion. When the material is capable of sustaining a tensile stress, there are stresses of this nature in region III. Any shearing stresses which may arise in this region must become developed as an internal resistance and are passive in character. They are primarily dependent on the weight of the material and the interlocking effect of the particles. When the ultimate carrying capacity is reached, the region I tends to burst region II which is supported by III. The resistance which region III offers to being pushed up is due to the coefficient of internal resistance, Rankine's passive pressure. For compact dry sand, failure takes place with an expansion in volume of the material in region III as well as in regions I and II.

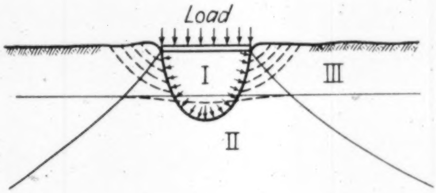

FIG. 1.—Sketch Showing the Regions of Stress in Granular Materials.

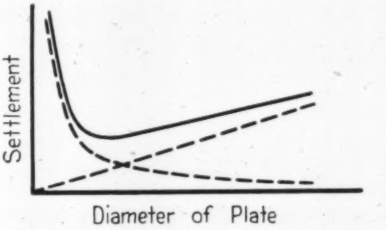

FIG. 2.—Relation of Settlement to Diameter of Plate (Kögler).

The term coefficient of internal resistance seems quite appropriate here. It is a function of a number of variables which assume rolls of greater or less importance according to the circumstances. Among these, the interlocking effect is of prime importance, as pointed out by the author. The accompanying Fig. 1 is intended as a modification of Fig. 1 of the paper. The writer prefers the bulk-like outline for region I because he believes it to be more nearly in accord with the natural phenomenon. As nearly as he can judge for dry sand confined between glass plates and loaded vertically, the shape is that of a round nosed rifle bullet. Horizontal layers of colored sand bend downwards through regions II and I as indicated and the effects appear to extend to a distance below the loaded area of approximately three times the width of that area.

Stability is a word often used in connection with earthwork, but it is also used more precisely in mechanics. One speaks of the stability of a mechanical system or of a structure. Under certain conditions a mechanical system may pass from the stable to the unstable state. The long column is the classical example of this. But it is a comparatively simple matter as compared with the stability of a mass of individual particles and it would seem expedient perhaps to qualify the expression as applied to granular masses for the sake of precision. The adjectives which suggest themselves are active and passive. It may become necessary to speak of "active stability" and "passive stability" to correspond to the active and passive pressures of Rankine. It is, of course, the passive stability which is implied in the paper.

It would be well, perhaps, to add emphasis to the warning that the "testing procedure must simulate actual conditions." In fact, more than mere simulation is necessary, for, as pointed out in the paper, scale effects are of prime importance. It will be difficult, if not impossible, to work out a theory of models. This has already been indicated by the fact that the conclusions arrived at from these tests, while quite correct when applied to bearing areas of comparable size, do not apply to bearing areas of much larger size. In *Die Bautechnik* for May, 1931, Heft 24, p. 358, Kögler presents some curves obtained experimentally for compacted sand. These show without doubt that, for circular plates less than 10 in. in diameter, and for constant unit load, the settlement is greater as the diameter of the plate decreases. This agrees with the author's findings. But on the other hand, for the same sand, Kögler finds that for larger plates, the settlement is greater as the diameter of the plate increases.

THE LOS ANGELES ABRASION MACHINE FOR DETERMINING THE QUALITY OF COARSE AGGREGATE

BY D. O. WOOLF[1] AND D. G. RUNNER[1]

SYNOPSIS

This paper presents the principal data considered in the development of a method of test for coarse aggregate using the Los Angeles abrasion machine, together with the results of an extensive series of tests in which the service behavior of materials used in surface treatment work are compared with the losses in Deval and Los Angeles abrasion tests.

Dissatisfaction with the standard Deval abrasion test for determining the quality of coarse aggregate led to the investigation of the Los Angeles abrasion test proposed by the California Division of Highways. Several features of the test method were changed to promote more uniform results and to adapt the method to the testing of materials used in low-cost road construction. As now presented, the method is believed suitable for use in determining the quality of practically all coarse aggregates used in road construction.

Service records for materials from 44 different sources are compared with the results of Deval and Los Angeles abrasion tests on samples from each source. A definite agreement seems to exist between the results of the Los Angeles test and the service record. The results of the Deval test show no agreement with the service record, and in a number of cases indicate material to be of satisfactory quality while the service record finds the material to be questionable or unsuitable for use.

Based on the tests reported, the authors find the Los Angeles abrasion test to be better suited for determining the quality of coarse aggregate than the standard Deval abrasion test. A percentage of wear of 40 in the Los Angeles test is suggested as a suitable specification limit for aggregate for use in surface treatment work.

The standard Deval abrasion test[2] and the standard toughness test[3] have been used for many years to control the quality of ledge rock for road work, although in the past few years they have been subject to considerable criticism. More recently a method based on the original Deval test but modified so as to make it possible to test a graded sample of rounded gravel has been issued by the Society.[4] The American Association of State Highway Officials has also modified the original Deval test in order to make it apply to light-weight material such as slag.[5] These several modifications, while necessary in order to adapt the Deval test to such materials as gravel and slag, also made necessary entirely different specification limits for the three classes of materials even when intended for the same service. An illustration of this is the current specification of one of the State highway departments. For grade A coarse aggregate for concrete a maximum wear of 6 per cent is allowed in the case of limestone, 15 per cent in the case of blast-furnace slag and 12 per cent in the case of rounded gravel, the requirements being intended to cover materials of comparable quality. The differences are necessary because of the differences in test values obtained in the respective tests on materials of comparable quality. Such a situation is, of course, unfortunate because of the apparent inconsistency in the use of different limits for different materials, all of which are to meet the same service requirements.

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[1] Associate Materials Engineer, and Assistant Materials Engineer, respectively, U. S. Bureau of Public Roads, Washington, D. C.

[2] Standard Method of Test for Abrasion of Rock (D 2 - 33), 1933 Book of A.S.T.M. Standards, Part II, p. 944.

[3] Standard Method of Test for Toughness of Rock (D 3 - 18), 1933 Book of A.S.T.M. Standards, Part II, p. 1006.

[4] Tentative Method of Test for Abrasion of Gravel (D 289 - 28 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 940 (1928); also 1934 Book of A.S.T.M. Tentative Standards, p. 708.

[5] A.A.S.H.O. Tentative Standard Method of Test for Abrasion of Broken Stone and Broken Slag (T-3), Tentative Standard Specifications for Highway Materials and Methods of Sampling and Testing, Am. Assn. State Highway Officials, p. 98 (1931).

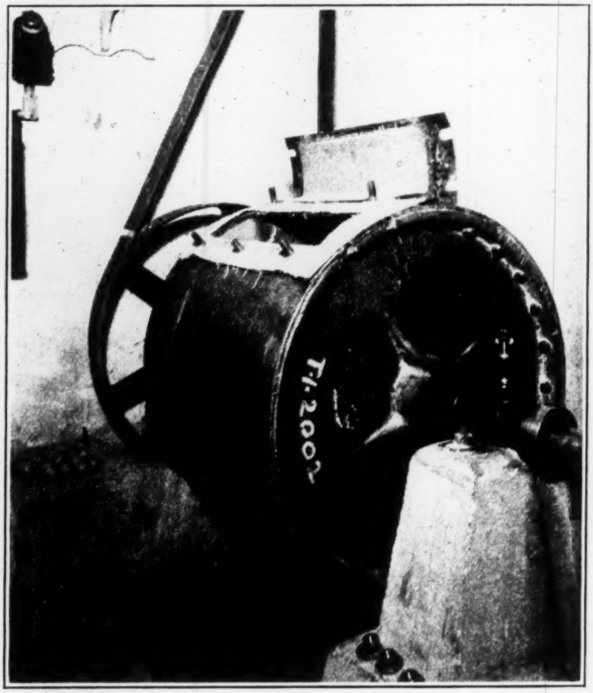

FIG. 1.—Los Angeles Abrasion Machine.

A criticism of the standard Deval abrasion test frequently made is the comparatively small range in values found in testing rock of the quality ordinarily used in road construction. The test is essentially an abrasion rather than impact test and for this reason certain types of materials which are very low in toughness but at the same time quite hard will show relatively low abrasion losses in this test. Certain granitic materials fall in this class. Such materials are frequently reported as giving unsatisfactory results in service, even though the percentage of wear by the Deval test may be quite low.

The standard toughness test has also received considerable criticism recently, principally from the standpoint of the accuracy of the test method itself. Attention has been called to the variations in results reported by different laboratories on apparently identical materials. Investigation indicates that the trouble is caused by the flattening of the spherical end of the plunger of the testing machine through use, with the resultant tendency to give higher values. Since the bulk of the test data upon which many of the State specifications for toughness were based was secured before the necessity for rigidly controlling this variable was appreciated, the matter becomes of considerable importance in connection with the acceptance of materials tested by a machine conforming strictly to the specification requirements as regards this detail. It is rendered all the more important by the fact that many sources of rock are strictly borderline materials from the standpoint of toughness so that even a small variation in results may mean the difference between acceptance and rejection.

The realization of these and other weaknesses in the present standard tests caused the Bureau of Public Roads recently to investigate the possibilities of the so-called "Los Angeles Rattler" test used by the State of California as an acceptance test for coarse aggregates.

THE LOS ANGELES ABRASION TEST

A number of years ago a machine for determining the abrasive resistance of aggregates was developed by the City of Los Angeles, Calif.[1] The method involved was radically different from the commonly used standard Deval abrasion test in that the test charge was caused to drop instead of slide or roll and, also, that an abrasive charge and a sample composed of graded sizes of particles were used. A test run of 500 revolutions was employed instead of the 10,000 revolutions required for the Deval machine, thus greatly reducing the time required for the operation of the test. In 1927, the California State Highway Laboratory made a study of the Los Angeles abrasion machine and apparently the machine and test method were found to be satisfactory for in that same year the test method was adopted by the State as a standard method. Some changes in the method as originally proposed were made, the latest (1930) test method being as follows:[2] "The machine used in the test consists of a cylindrical drum 28 in. in diameter and 20 in. in length, mounted longitudinally on a horizontal shaft, and having a shelf 4 in. wide extending from end to end on the inside.

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[1] C. L. McKesson, "Selection of Rock and Gravel for Highway Construction," *California Highways*, Vol. 3, No. 4, April, 1926, pp. 6–11.

[2] From unpublished report of California Division of Highways, dated June 13, 1930.

"The drum is charged with 14 cubical blocks of cast iron having rounded corners and edges and weighing a total of 5000 g. along with 5000 g. of rock, which is graded as follows:

SCREEN SIZE, IN.	TOTAL PASSING, PER CENT
1½	100
1¼	80
1	60
¾	40
½	0

"After charging, the drum is revolved 100 revolutions and 500 revolutions at a rate of between 30 and 33 revolutions per minute. The result is reported as the percentage of wear at 100 and 500 revolutions. At the present time the wear is considered that portion of the sample which, after test, will pass a 10-mesh sieve having a clear opening of 0.065 in. (No. 12 U. S. Standard)."

In its report the California Division of Highways cites certain advantages possessed by this method of testing, as follows:

"The Los Angeles rattler test is decidedly more suitable for determining the hardness and toughness of rock and the amount of soft material than any test or group of tests studied. Its advantages are pointed out as follows:

"1. The nature of the treatment is severe, bringing out weaknesses not shown by any one of the other tests studied.

"2. It is adapted for testing both crushed and gravel aggregates.

"3. It requires very little time for performance.

"4. It is not affected materially by changes in volume of aggregate due to specific gravity because of the size of cylinders in which the test is made.

"5. It eliminates a large amount of the personal equation which enters into some of the other tests."

In order to determine whether the conclusions reached by California would apply to tests covering a wider range in materials a study of the test method was undertaken by the Bureau. A machine was constructed according to plans furnished by the California Division of Highways, as shown in Fig. 1. The shelf which picks up the charge is mounted on the removable cover. This cover was originally fastened on by two bolts at each end. A few tests showed that the cover tended to spring at the center, allowing dust to escape, and to prevent this the cover is now fastened by two heavy bars, curved to fit the drum and fitting over the stud bolts projecting from the drum. The gasket consists of four thicknesses of heavy canvas firmly sewed together.

Samples of rock, gravel and slag from various parts of the country were obtained, largely from the producers, for the tests. Where possible each sample of rock was composed of both ledge rock and crushed material.

The producers had been requested to furnish the crushed and ledge rock of the same quality and as far as could be observed, this was done. These samples represent practically all types of rock and gravel used for road building. Although only three samples of slag were tested these represent the 70 to 85-lb. class of blast-furnace slag widely used in highway construction.

INVESTIGATION OF ABRASIVE CHARGE

Prior to the principal series of tests a preliminary study was made to determine the possibility of substituting balls for the cubical shot used by California. The procedure followed by California specified an abrasive charge of fourteen $1\frac{1}{2}$-in. cast iron cubes. In the standardization work on the brick rattler, abrasive charges of both cubes and balls were tried and balls were finally adopted.[1] The cast iron balls proposed for use in the Los Angeles machine were the same as the small balls in the brick rattler machine which have a nominal diameter of $1\frac{7}{8}$ in. and an initial weight of about 0.95 lb. Since twelve new balls would weigh about 5170 g., the stock of used balls from the brick rattler was inspected, and twelve balls were selected which weighed 5000 ± 5 g. To insure representative results in the preliminary series of tests, one sample of each of the three materials, rock, gravel, and slag, was selected for test and at least nine samples of each material were tested with each type of abrasive charge. Determinations of the percentage of wear were made at the end of 100 and 500 revolutions. At 100 revolutions the material was taken from the machine, sieved on a No. 12 sieve, and the particles brushed free from adhering dust.

TABLE I.—COMPARISON OF CUBES AND BALLS AS ABRASIVE CHARGE IN LOS ANGELES MACHINE.

Sample	Kind of Material	Abrasion Loss, per cent			
		Cubes		Balls	
		100 rev.	500 rev.	100 rev.	500 rev.
No. 33265	Slag	8.2	38.3	9.7	46.7
No. 33278	Gravel	13.7	52.0	17.7	59.3
No. 33279	Basalt	1.5	6.1	2.0	9.7

After the amount passing the sieve had been determined, the entire charge including the dust was replaced in the machine and the test resumed for an additional 400 revolutions. The results of these tests are shown

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[1] M. W. Blair and Edward Orton, Jr., "A Study of the Rattler Test for Paving Brick," *Proceedings*, Am. Soc. Testing Mats., Vol. XI, p. 776 (1911).

TABLE II.—LOS ANGELES ABRASION TEST SAMPLE. GRADING A.

Screens with round openings.

PASSING	RETAINED ON	WEIGHT, G.
1½ in.	1¼ in.	1000
1¼ in.	1 in.	1000
1 in.	¾ in.	1000
¾ in.	½ in.	2000
Total		5000

TABLE III.—RESULTS OF ABRASION AND TOUGHNESS TESTS.

Sample	Kind of Material	Location	Wear, per cent			Toughness
			Deval Test	Los Angeles Test		
				Gravel, Crushed Rock, Slag	Hand-Broken Rock	
No. 33262	Slag[a]	Pa.	7.8	34.8	....	....
No. 33263	Aplite	Md.	2.0	18.4	15.9	16
No. 33264	Limestone	W. Va.	3.8	29.2	24.2	6
No. 33265	Slag[b]	Ohio	14.8	46.7	....	....
No. 33266	Gravel	N. J.	15.1[c]	49.8	....	....
No. 33269	Dolomite	Ohio	5.8	37.0	30.7	5
No. 33270	Biotite granite	S. C.	2.5	27.7	21.9	12
No. 33271	Quartzite	Wisc.	2.6	25.6	19.6	12
No. 33272	Argillaceous limestone	Kans.	3.5	30.5	26.4	6
No. 33273	Diabase	N. J.	1.5	13.5	8.3	20
No. 33274	Dolomite	Ill.	5.9	28.4	26.1	6
No. 33275	Gravel	Wash.	1.1[d]	10.4	....	....
No. 33276	Slag[e]	Ohio	11.8	36.9	....	....
No. 33278	Gravel	W. Va.	10.0[d]	47.5[f]	....	....
No. 33278	Gravel	W. Va.	....	59.3[g]	....	....
No. 33279	Basalt	Minn.	1.8	9.7	7.0	32
No. 33992	Gravel	D. C.	8.4[d]	30.3[f]	....	....
No. 33992	Gravel	D. C.	17.5[h]	35.3[g]	....	....
No. 34165	Limestone	Kans.	14.4	43.0	....	3
No. 34542	Gravel	Kans.	7.5[d]	17.6	....	....
No. 34543	Argillaceous limestone	Kans.	12.8	42.9	....	4
No. 34544	Dolomite	Ohio	23.6	98.5	....	2
No. 34545	Gravel	Ohio	13.8[d]	39.3	....	....
No. 34571	Dolomite	Ohio	8.2	49.2	....	4
No. 34572	Gravel	Ohio	7.2[d]	27.3	....	....
No. 34671	Marble	Ga.	6.2	58.3	....	3
No. 34672	Marble	Ga.	5.4	50.4	....	4
No. 34673	Marble	Ga.	12.8	72.2	....	3
No. 34674	Marble	Ga.	12.0	74.4	....	3
No. 34675	Dolomitic marble	Ga.	7.2	45.5	....	3
No. 34676	Dolomitic marble	Ga.	5.0	39.2	....	....
No. 34685	Limestone	Fla.	6.7	35.6	....	....
No. 34700	Gravel	Va.	13.7[d]	45.1	....	....
No. 34701	Gravel	Md.	18.6[d]	53.7	....	....
No. 34704	Micaceous granite	Ga.	4.8	67.4	....	....
No. 34713	Gravel	Va.	13.0[d]	43.8	....	....
No. 34714	Gravel	N. Y.	7.6[d]	42.1	....	....
No. 34715	Argillaceous limestone	W. Va.	4.0	23.4	....	....
No. 34717	Limestone	Ohio	4.5	22.0	....	....
No. 34722	Dolomite	Ohio	8.6	37.3	....	....
No. 34723	Argillaceous dolomite	Ohio	3.7	24.4	....	....
No. 34724	Gravel	Ill.	5.3[d]	33.2	....	....
No. 34732	Dolomite	Va.	3.9	21.5	....	....
No. 34733	Limestone	Ohio	8.2	62.8	....	....
No. 34734	Argillaceous dolomite	Ohio	3.9	20.8	....	....
No. 34749	Gravel	Pa.	5.5[d]	29.3[f]	....	....
No. 34750	Gravel	Pa.	15.2[h]	34.6[g]	....	....
No. 34756	Gravel	Mass.	17.9[d]	46.8	....	....

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[a] Weight per cubic foot, 80.7 lb.

[b] Weight per cubic foot, 72.2 lb.

[c] Grading B, rounded particles only (See A.S.T.M. Method D 289 - 28 T).

[d] Grading A, rounded particles only.

[e] Weight per cubic foot, 77.0 lb.

[f] Rounded particles only.

[g] Angular particles only.

[h] Grading A, angular particles only.

in Table I. It will be observed that in all cases the balls furnished a greater loss in abrasion than the cubes. The greater weight per unit of area of the ball together with the delivery of impact at a single point caused greater loss in the test than the edges and corners of the cube.

Besides furnishing the more severe action on the test specimen, the abrasive charge of balls gives slightly more concordant results. Tests with the ball abrasive charge show an average deviation of 3.4 per cent in the loss at 500 revolutions, while the corresponding average deviation for the cubes is 5.5 per cent. More concordant results were obtained at 500 revolutions than at 100.

The cube was found to lose weight at a much more rapid rate than the ball. This required constant adjustment of the cube abrasive charge and possibly affected the abrasive loss. The ball lost very little weight after the first few tests. This feature, combined with the greater action on the test sample and the more concordant results obtained, shows that the ball is preferable for use in the abrasive charge. Furthermore, many laboratories are equipped with the brick rattler and presumably have a supply of the cast iron balls of suitable size and weight.

TESTS OF AGGREGATES—SERIES A

With the decision to adopt the use of balls as the abrasive charge, the main portion of the investigation was considered. It was desired to compare the results of the Los Angeles abrasion test with those for the Deval abrasion test and, also, to determine the effect of angularity of particle on the Los Angeles test results. Each sample was tested in the Deval and Los Angeles abrasion machines and toughness tests were made on all suitable samples of rock. The Deval abrasion tests on rock and slag were made in accordance with A.S.T.M. Standard Method D 2 – 33, and those on gravel in accordance with A.S.T.M. Tentative Method D 289 – 28 T. The Los Angeles tests followed the procedure used by California with the exception that the abrasive charge consisted of twelve 1⅞-in. cast iron balls weighing 5000 ± 5 g. To determine the effect of shape and angularity of particles on the test result, tests were made on commercially crushed rock and hand broken pieces of cubical shape with sharp edges and corners and, also, on both rounded and angular particles of gravel and rock.

In preparing the test specimens for the Los Angeles test the samples of gravel, slag, and rock in both crushed and hand-broken condition were separated into the various screen sizes and recombined as shown in Table II.

The results of the standard Deval abrasion tests and the Los Angeles tests using grading A are given in Table III. Most of the abrasion test values given are averages of three or more tests. These values have also been plotted in Fig. 2 together with curves showing the average relation between the standard and modified Deval tests and the results in the

Los Angeles machine. The number of points which depart from the average curves demonstrate that the relations are only very general and do not apply to all types of materials nor to all samples of a given type. This is due mainly to the marked difference in the amount of impact produced in the two tests. Although both tests involve both surface wear and impact, the loss in the Deval test is mainly from surface wear, while that for the Los Angeles test is primarily caused by impact. Marble, for example, has about the same wear in the Deval test as limestone or dolomite but in the Los Angeles test, the marble shows a much higher loss than the tougher rocks. Figure 2 is, accordingly, presented with the knowledge that no definite relationship between the losses in the Deval and Los Angeles tests can be established which will apply to all materials. The figure will, however, serve to show the approximate change in specification requirements if the Deval test is replaced by the Los Angeles test. For instance, it will be observed that, for an average loss of 50 per cent in the Los Angeles test, the average Deval abrasion loss is about 7.0 per cent in the case of rock and 15.0 per cent in the case of gravel. This latter ratio is approximately the same (1:2) as is used in most specifications with the idea of obtaining materials of comparable quality.

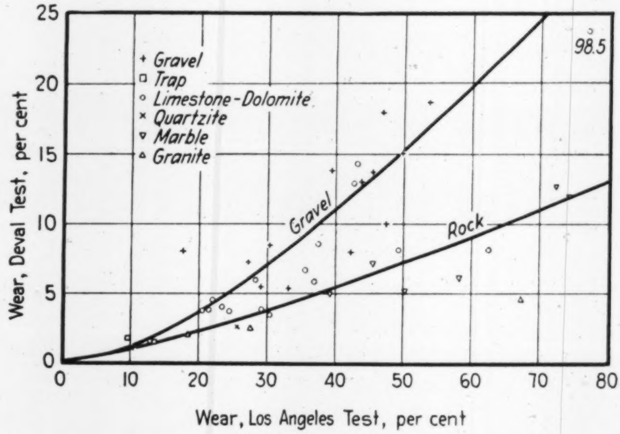

FIG. 2.—Relation Between Results of Tests in Deval and Los Angeles Machines.

More concordant results are obtained in tests of gravel and crushed rock or slag in the Los Angeles machine than in tests using the Deval abrasion machine. Tests in the Los Angeles abrasion machine show a mean variation from the average of 2.7 per cent, while those in the Deval machine have a mean variation of 3.9 per cent. The difference is not great but it is worthy of notice. It was observed that the speed of operation of the Los Angeles abrasion machine had a great effect on the loss during the test and it was found advisable to equip the motor with a speed control. Each test run was timed to insure that a constant speed of rotation had been obtained.

EFFECT OF SHAPE AND ANGULARITY OF PARTICLE

In its report on the Los Angeles abrasion machine, the California Division of Highways states that the test results are not appreciably affected by the shape or angularity of the particles. The results obtained in the tests reported here are not entirely in agreement with this statement. To investigate this feature the samples of Cheat River, Potomac River, and Delaware River gravels were carefully hand picked and the rounded and angular particles separated. Tests were made in the Los Angeles abrasion machine with both kinds of particles with the results given in Table IV.

TABLE IV.—TESTS ON ROUNDED AND ANGULAR GRAVEL IN LOS ANGELES MACHINE.

Each value is the average of at least 3 tests.

Sample	Material	Wear, per cent		
		Rounded	Angular	Ratio, Angular to Rounded
No. 33278	Cheat River gravel	47.5	59.3	123
No. 33992	Potomac River gravel	30.3	35.3	117
No. 34749 No. 34750	Delaware River gravel	29.3	34.6	118

TABLE V.—TESTS ON ROUNDED AND ANGULAR ROCK IN LOS ANGELES MACHINE.

Each value is average of three tests.

Sample	Material		Wear, per cent		
	Kind	Type	Rounded	Angular	Ratio, Angular to Rounded
No. 34549	Limestone	Crushed	31.4	33.4	106
No. 34631	Sandstone	Hand broken	62.8	64.4	103
———	Limestone	Hand broken	22.4	23.9	107

It is stated that in the preparation of the Cheat River and Delaware River gravels for use as concrete aggregate the oversize material is crushed. It is quite possible that the majority of the angular particles used in our tests came from this oversize material. Visual inspection, however, failed to show any marked difference in quality between the rounded and angular fragments. The Potomac River gravel sample did not contain crushed material. The angular particles obtained from this material were distributed throughout the entire range in size and have the same petrographic analysis as the rounded particles. The tests of these three gravels indicate that angular particles will give a somewhat higher loss than rounded particles of the same quality. Tests of hand-broken and crushed rock shown in Fig. 3, however, demonstrate that the shape of the particle exerts a considerable influence on the test result, and that the partially wedge-shaped fragment of crushed rock has a loss of approximately 120 per cent of that for the hand-broken fragment of cubical shape. In the tests of the three gravels, the rounded samples contained a greater proportion of particles which tended toward being spherical and offered more resistance to impact than the samples containing the angular particles. It is reasonable to apply the findings of the crushed and hand-broken rock tests to these tests of gravel due to the difference between the shape of the particles of rounded and angular gravel. On this basis, a sample of angular gravel would be expected to give a loss of approximately 120 per cent of that found for a sample of rounded gravel of the same quality. It will be observed that approximately this same ratio was obtained in the tests of the three samples of gravel referred to above.

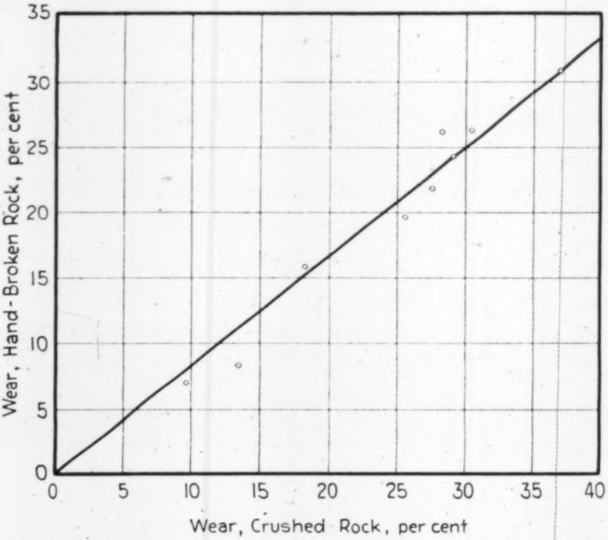

FIG. 3.—Relation Between Results of Tests on Hand-Broken (Cubical Piece) and Crushed Rock in Los Angeles Machine.

Further tests to determine the effect of shape and angularity were made on samples of angular and artifically rounded rock. One large sample of crushed rock and two of hand-broken rock were obtained, and a portion of each was run in the Deval abrasion machine until the sharp edges and corners had been worn off. Duplicate test samples were then prepared from each material, one sample containing fragments with sharp edges and corners while the second was composed of rounded fragments. Each sample was tested in the Los Angeles machine with the results shown in Table V.

The angular samples show a slightly higher loss than the rounded samples, but in no case is the increase in wear similar to that found in the tests of gravel or crushed and hand-broken rock. It is apparent that while the sharpness of edge and corner may have some influence on the loss in the Los Angeles test, the shape of the particle exerts a much greater effect and for most purposes the angularity of the sample may be ignored.

Samples of aggregates containing large percentages of flat or elongated fragments will show a much higher percentage of wear than other materials of equal hardness in which the fragments tend more toward cubical shape.

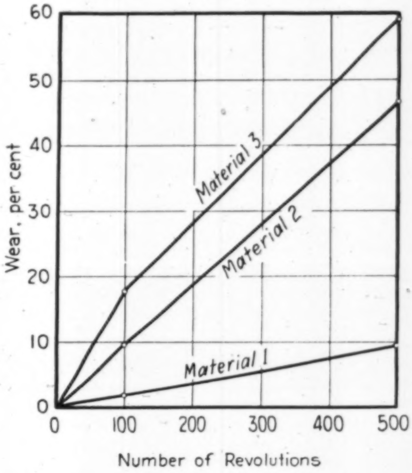

Fig. 4.—Relation Between Length of Test and Percentage of Wear, Showing Effect of Soft Particles in Material.

This discrimination between fragments of different shape agrees with the desire of highway engineers to limit the percentage of flat and elongated particles in aggregates. The relatively small effect of sharpness of edge and corner will permit the use of a single specification limit for crushed stone and either rounded or angular gravel.

Comparative Losses at 100 and 500 Revolutions

The method of test used by California requires the determination of the percentage of wear after both 100 and 500 revolutions. The determination after 100 revolutions is expected to show the presence of soft particles in the material. This can be found provided a sample of uniform composition shows a straight-line relation between loss and number of revolutions. To determine whether this is the case, reference is made to the preliminary tests made with cast iron balls shown in Table I. Figure 4 presents the relationship between the length of test and the loss. It will be seen that materials 1 and 2 have a straight-line relationship, that is, the loss varies directly with the number of revolutions. Material 3, however, is of non-uniform hardness since the loss at 100 revolutions is proportionately greater than that at 500 revolutions.

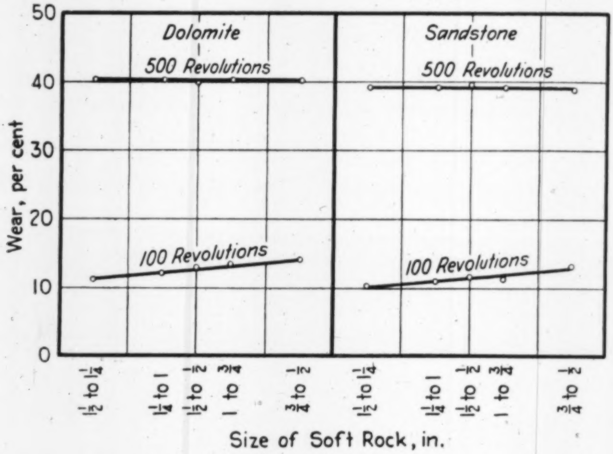

FIG. 5.—Effect of Size of Soft Rock Fragments; Each Sample Contained 10 per cent by Weight of Soft Rock.

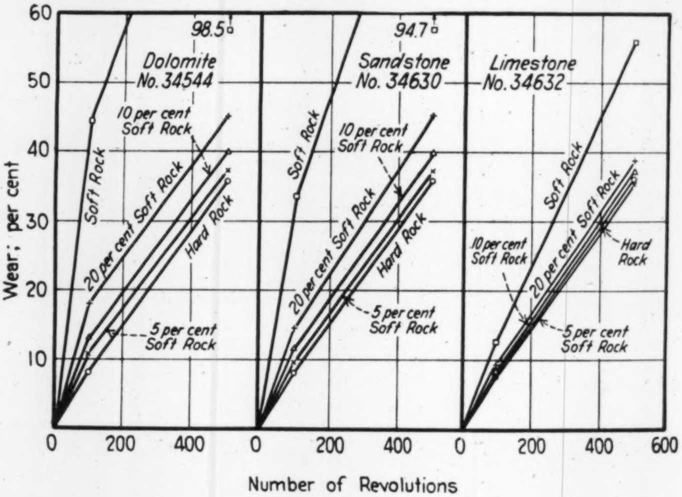

FIG. 6.—Effect of Amount of Soft Rock in Sample.

Tests to determine the effect of known amounts of soft rock in the sample are shown in Table VI and Figs. 5 and 6. A hard limestone of uniform composition was used as the base material, and soft rock in varying amounts and sizes was added to it to determine the effect on the percentage of wear. In the first series of tests the amount of soft rock in each sample was held constant at 10 per cent of the total weight of the sample while the size of the fragments of soft rock was varied. Tests were made with the soft rock contained entirely in each size of the test sample and also with the soft rock distributed in size from 1½ to ½ in. in the same proportions as is specified for the total sample. Determinations of the percentage of wear were made at 100 and 500 revolutions. As shown in Fig. 5, the loss at 100 revolutions is affected slightly by the size of the soft rock fragments, the loss increasing with reduction in size. At 500 revolutions, however, the size of the soft fragments has no apparent effect on the percentage of wear.

TABLE VI.—EFFECT OF SIZE AND AMOUNT OF SOFT ROCK ON ABRASIVE LOSS.

Composition of Sample, per cent		Soft Rock		Wear, per cent	
Hard	Soft	Type	Size, in.	100 rev.	500 rev.
FIRST SERIES					
90	10	Dolomite	1½ to 1¼	11.2	40.3
90	10		1¼ to 1	12.1	40.2
90	10		1 to ¾	13.3	40.2
90	10		¾ to ½	13.9	40.1
90	10		1½ to ½	13.0	39.9
90	10	Sandstone	1½ to 1¼	10.1	39.1
90	10		1¼ to 1	10.9	38.9
90	10		1 to ¾	11.3	39.0
90	10		¾ to ½	13.1	38.8
90	10		1½ to ½	11.4	39.4
SECOND SERIES					
100	0	None		8.0	35.7
95	5	Dolomite	1½ to ½	10.7	37.2
90	10		1½ to ½	13.0	39.9
80	20		1½ to ½	18.0	45.0
0	100		1½ to ½	44.0	98.5
95	5	Sandstone	1½ to ½	9.5	36.9
90	10		1½ to ½	11.4	39.4
80	20		1½ to ½	14.6	45.0
0	100		1½ to ½	33.6	94.7
95	5	Limestone	1½ to ½	8.0	35.4
90	10		1½ to ½	8.6	37.0
80	20		1½ to ½	9.0	38.9
0	100		1½ to ½	12.3	55.4

A second series of tests was made to determine the effect of varying the quantity of soft rock. The results are plotted in Fig. 6. The soft rock was distributed in all sizes of each sample in proportion to the amount of each size in the total sample, and soft rock amounting to from 5 to 20 per cent of the total weight of the sample was used. Tests were also made on samples composed entirely of hard or soft rock. Test of the hard rock sample gave a practically straight-line relationship between percentage of wear and number of revolutions. As soft rock was added, the loss at 100 revolutions increased at a more rapid rate than that at 500 revolutions, and the curve assumed a characteristic hump denoting a material of non-uniform hardness. It is of interest to note that the actual loss under test of a mixture of hard and soft rock agrees fairly well with a weighted loss computed from the percentages of wear for the separate materials.

It does not appear possible to determine the percentage of soft rock in a test sample entirely by inspection of the results of the 100 and 500 revolution test. A study of Fig. 6 shows that the difference in slope of the lines from zero to the 100 revolution point and between the 100 and 500 revolution points may be used to indicate the relative effect of soft rock in the test sample. This difference in slope increases with increase in the amount of soft rock present. It is then possible to determine whether the sample under test contains soft rock, but whether the adulterating material consists of a small amount of very soft rock or a large amount of relatively harder rock cannot be stated with the present method of test. A general indication of the uniformity of the sample is presented and in certain cases this may be of considerable interest. It is possible that some difference may be found by determination of the loss at some other period of the test than 100 and 500 revolutions, or that a complete mechanical analysis of the sample after testing will show the adulterating material more clearly.

TESTS OF AGGREGATES—SERIES B

During the winter of 1933, considerable attention was given by a number of research organizations to the problem of devising a satisfactory method of determining the quality of crushed material proposed for use in road surfacing where the aggregate is subjected directly to the action of traffic. Reports received from a number of different sources stated that the results of the Deval abrasion test bore no relation to the service record of materials used in surface treatment or other types of low cost road construction. The extensive use of these types of construction necessitated the development of a test which would indicate the suitability of materials for use in this work. One type of test proposed for this determined the resistance of crushed and graded material to the crushing caused by the passage of a heavy roller over a thin layer of the material.[1] Although

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[1] A simple and inexpensive machine for this test is described in a summary of research activities by State-highway departments, *Rock Products*, Vol. 34, No. 2, January 17, 1931, pp. 51–57. A more elaborate apparatus is described by A. T. Goldbeck, J. E. Gray and L. L. Ludlow, Jr., in "A Laboratory Service Test for Pavement Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 608 (1934).

TABLE VII.—LOS ANGELES ABRASION TEST SAMPLE.

Size, Square Opening, in.	Total Percentage Retained	
	Grading B	Grading D
1½	0	...
1	25	...
¾	50	0
½	75	50
⅜	100	100

Abrasive Charge:	GRADING B	GRADING D
Number of 1⅞-in. balls	12	11
Weight, g	5000 ± 5	4583 ± 5

TABLE VIII.—COMPARISON OF LOS ANGELES AND DEVAL ABRASION RESULTS WITH SERVICE BEHAVIOR OF MATERIALS.

Sample	Location	Kind of Material	Wear, per cent				Reported Service Record
			Deval Test	Los Angeles Test			
				Grading A	Grading B	Grading D	
No. 40110	Wis.	Altered basalt	....	....	....	10.5	Satisfactory
No. 40280	Va.	Argillaceous dolomite	2.9	....	17.2	20.9	
No. 40279	Va.	Aplitic granite	1.7	....	17.4	19.2	
No. 40125	Ohio	Argillaceous limestone	7.3	....	20.2	26.0	
No. 40276	Va.	Amphibolite	1.7	....	23.0	22.8	
No. 40178	N. Y.	Argillaceous limestone	3.9	....	24.8	24.3	
No. 40084	Ga.	Dolomite	3.6	....	25.2	....	
No. 40108	Wis.	Dolomite	3.2	....	25.3	24.6	
No. 40119	Pa.	Quartzite	4.4	....	26.5	24.6	
No. 40149	Tenn.	Argillaceous limestone	3.0	....	26.6	25.0	
No. 40059	Ga.	Limestone	3.6	27.2	....	....	
No. 40231	N. Y.	Argillaceous limestone	4.0	....	28.0	25.4	
No. 40086	Ala.	Slag	10.5	....	28.2	....	
No. 40107	Wis.	Gravel	6.6	....	29.1	28.9	
No. 40109	Wis.	Dolomite	3.3	....	30.1	30.0	
No. 40138	Tenn.	Limestone	3.0	....	30.2	29.4	
No. 40182	N. Y.	Cryst-argillaceous limestone	4.9	....	....	30.6	
No. 40277	Va.	Argillaceous limestone	3.5	....	31.1	30.7	
No. 40120	Ill.	Dolomite	....	....	31.3	29.6	
No. 40067	Ga.	Slag	10.5	31.6	....	....	
No. 40274	Va.	Granite	2.6	....	32.7	33.4	
No. 40180	Mich.	Limestone	3.4	....	33.7	33.8	
No. 40111	Wis.	Dolomite	4.1	....	34.4	33.0	
No. 40136	Tenn.	Limestone	2.8	....	34.6	32.4	
No. 40229	N. Y.	Argillaceous limestone	4.9	....	34.8	....	
No. 40137	Tenn.	Gravel	....	....	34.1	37.7	
No. 40147	S. C.	Granite	3.8	....	35.0	36.0	
No. 40275	Va.	Granite	1.8	....	36.6	37.6	
No. 40106	Wis.	Gravel	13.4	....	....	37.5	
No. 40112	Wis.	Argillaceous limestone	6.3	....	39.4	35.6	
No. 40098	Okla.	Limestone	....	....	40.4	39.4	
No. 40085	Ga.	Dolomitic marble	6.0	....	41.9	....	Questionable
No. 40194	N. Y.	Biotite gneiss	4.2	....	47.9	39.5	Satisfactory
No. 40278	N. C.	Granite	1.9	....	41.0	43.6	Questionable
No. 40281	Va.	Granite	1.9	....	41.8	46.9	
No. 40114	Md.	Dolomitic marble	4.1	....	47.0	45.4	
No. 40057	Ga.	Granite	3.5	47.7	....	....	
No. 40146	S. C.	Gravel	12.4	....	50.0	51.0	
No. 40430	Tenn.	Limestone	8.5	....	41.4	42.0	Not suitable
No. 40164	Mich.	Limestone	5.7	....	53.0	51.0	
No. 40126	Ohio	Limestone	36.8	....	54.8	52.2	
No. 40056	Ga.	Granite	2.8	65.2	....	....	
No. 40145	S. C.	Gneissoid granite	4.8	....	67.6	....	
No. 40144	S. C.	Gravel	33.6	....	83.0	....	

excellent results were reported with the roller test, the labor and time required rendered it unsuitable as an acceptance test and it was suggested that possibly the less involved Los Angeles abrasion test would furnish equally good results.

It was realized that in order to cover the range in size of material used in surface treatment work, provision should be made for testing aggregate with a maximum size of about $\frac{3}{4}$ in. In other types of low cost road improvement the aggregate has a maximum size of about $1\frac{1}{2}$ in., and it was decided to secure gradings of test samples which would permit testing both sizes of aggregate. When these gradings were being considered, the screens used in preparing the test samples were changed to sieves with square openings to agree with the growing trend in the use of this shape of opening for road materials.

After considerable experimentation, two gradings for the Los Angeles abrasion test were adopted which were believed to be suitable for testing practically any size of material used in bituminous or concrete pavements. It was found that if the abrasive charge was changed slightly with the smaller grading, the loss for both gradings would be approximately the same. This would permit the establishment of a single specification limit for a material irrespective of the grading used in the test. The gradings and abrasive charges finally adopted are given in Table VII.

Some concern was expressed that with the change from the previously used grading A (round openings) to gradings B and D; the relationships established for grading A could not be applied to the other gradings. However it was found that the losses for grading B were so nearly the same as those for grading A that for all practical purposes the established data could be applied to test results for grading B.

In order to obtain definite information regarding the significance of the Los Angeles abrasion test in terms of service behavior, samples of crushed rock, gravel, and slag which had been used in surface treatment construction were obtained from a number of state highway departments. Tests of these materials were made in the Los Angeles abrasion machine using gradings A or B and D when possible. In the majority of cases, at least three tests were made with each grading. The results of these tests are shown in Table VIII. In Fig. 7 a very close agreement is found between the average result of the Los Angeles abrasion test and the service record of the materials. With the exception of one sample, all materials found to be of satisfactory quality in service show losses in the Los Angeles abrasion test of 40 per cent or less. Materials of questionable suitability show losses between 40 and 50 per cent, while with one exception materials which had been found to be unsuitable for use have losses of over 50 per cent.

No relationship is found between the service record and the loss in the

Deval abrasion test. Of the 10 samples of rock reported as questionable or unsatisfactory, 8 have percentages of wear of 6 or less and would be con-

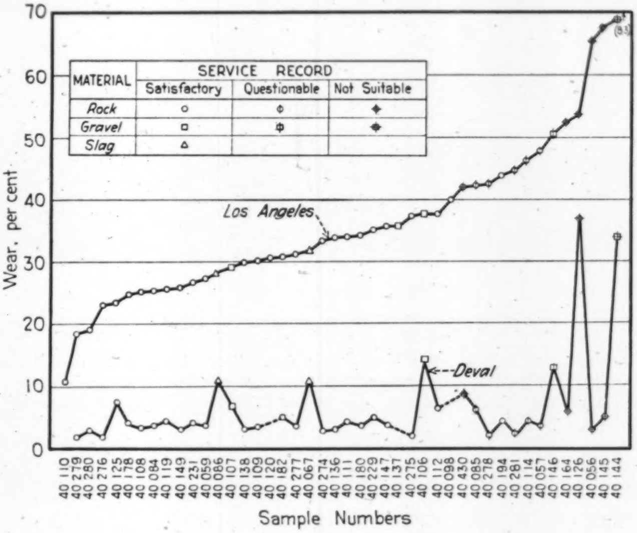

FIG. 7.—Comparison of Abrasion Test Results with Service Record.

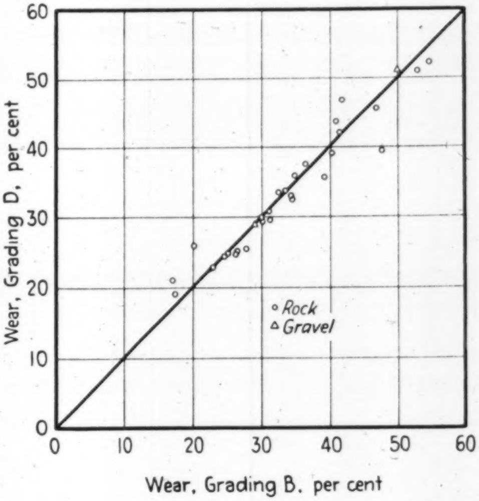

FIG. 8.—Relation Between Losses for Gradings B and D—Los Angeles Abrasion Test.

sidered suitable for use under many present specifications. The one sample of gravel which is reported as questionable has a Deval loss of 12.4 per cent and would also probably be accepted for use on the basis of present standard methods of testing coarse aggregates. The great difference in the ability of the Los Angeles and Deval tests to show clearly the quality of coarse aggregate is well illustrated in Fig. 7. As an example, samples 40279 and 40281 have the same percentage of wear in the Deval test but in the Los Angeles test the latter material has over twice the loss of the former.

In all, comparisons between the loss in the Los Angeles abrasion test and the service record were made on materials from 44 different sources. The remarkable concordance of the results permits the belief that these tests furnish sufficient data to warrant the tentative establishment of a loss in the Los Angeles abrasion test of 40 per cent as an acceptable limit for material which will prove satisfactory for use in surface treatment work.

The samples tested by both gradings B and D have in general a very good agreement between the percentages of wear for either grading. As shown in Fig. 8, in only five cases does the loss for one grading differ by more than 3 per cent from that for the other. In the case of three of the five samples, the loss for grading D is the greater, and it is believed that in the crushing operations the softer rock tended to be reduced in size to a greater extent than the harder rock in the quarry. This would account for the greater loss of the finer of the two gradings. The other two samples, a biotite gneiss and an argillaceous limestone, contain an appreciable percentage of flat fragments of rock. The excessive loss for grading B is attributed to these flat fragments which are found to a greater extent in the larger sizes of the sample. Considering all samples tested, the average difference between the losses for gradings B and D is only 1.9 per cent. It is believed that either grading may be used in acceptance or control tests of coarse aggregates.

CONCLUSION

The results of this investigation demonstrate that the Los Angeles abrasion machine is superior to the present standard Deval machine in the following respects:

1. Tests can be made much more rapidly and more accurately.
2. Both round and angular particles may be tested with very little difference in percentage of wear due to the degree of angularity.
3. The Los Angeles abrasion test result is greatly affected by the shape of the particles under test. Thus the presence of flat or elongated fragments in a sample under test would increase the loss in the Los Angeles test, while in the Deval test these possibly objectionable particles might have little effect on the percentage of wear.
4. The presence of soft or friable rock can be detected with the Los Angeles test but not with the Deval abrasion test.
5. A definite agreement seems to exist between the loss in the Los Angeles abrasion test and the service record of materials used in surface

treatment construction. Based on the results available to date, materials having a loss in the Los Angeles abrasion test of 40 per cent or less may be expected to furnish satisfactory results when used in surface treatment construction.

6. Differences in the volume of different test samples due to changes in specific gravity need not be considered, due to the relatively large volume of the Los Angeles abrasion machine.

7. Dust produced in the test does not affect the result as is found to be the case in the Deval test.

8. The Los Angeles test is made on material as prepared for use on the project, while the Deval test for rock requires the use of ledge rock which may not represent the material actually used. The two gradings, B and D, proposed for use in the Los Angeles abrasion test furnish practically the same result, and specification tests may be made using the grading which can most readily be prepared from the material submitted for test.

9. The effect of personal equation in the preparation of the test sample is largely eliminated.

A disadvantage of the Los Angeles test is that no provision is made for testing ledge rock taken from undeveloped quarries. However, as shown in Fig. 3, a fairly definite relationship exists between samples of crushed and hand-broken rock, and tests could be made on the ledge rock provided the result is corrected to agree with those for the crushed material.

DISCUSSION

MR. T. A. FITCH[1] (*by letter*).—The Los Angeles abrasion test and equipment originated in the laboratories of the Bureau of Standards of the City of Los Angeles and has been specified and used continuously in street construction work in Los Angeles for 18 yr.

It is true that this test does not detect the presence of small amounts of very soft rock. Therefore the following requirement preliminary to the abrasion test is incorporated in the specifications of this city:

> *Unsound Material.*—Rock shall not contain more than 3 per cent by weight of pieces that can be crumbled with the fingers, either dry or after being immersed in water for 18 hr. and then dried.

MR. A. T. GOLDBECK[2] (*presented in written form*).—The paper presented by Messrs. Woolf and Runner promises to be of high importance in the field of testing of aggregates. Although there has been a radical change in methods of highway construction and a still more radical alteration in highway vehicle and tire design, extending over the past twenty years, highway engineers have continued to specify the quality of aggregates in terms of physical tests which were devised originally with waterbound macadam and horse-drawn vehicles in mind.

Tests for quality of materials are apt to be very misleading unless it is proven that they have a relation to the behavior of those materials in service. Fortunately, the authors of the present paper have recognized that fact and have taken the trouble to correlate their test results with the observed behavior of the materials under the action of traffic. They seem to have obtained an exceedingly good indication that the Los Angeles rattler test can be relied upon to indicate the quality of aggregates for use in certain types of highway construction, particularly for aggregates used as cover material in bituminous surface treatment work and for use also in the more or less open types of cold laid bituminous concrete. Because of this correlation, test values may be set with at least a reasonable degree of certainty that they will exclude unsuitable materials and include those which are suitable.

Two of our time-honored tests, namely, the Deval abrasion test and the standard test for toughness, seem to be not as suitable for judging the quality of aggregates for the above mentioned types of construction as is the rattler test described by the authors. At the last annual meeting of the Society, the speaker presented a paper[1] in a part of which there was described a test made by the use of a roller for determining the resistance of various rocks to crushing. The roller acts in the same manner as a roller in service and, consequently, the test described is in effect a service test. It will be interesting therefore to compare the results of this roller test or service test with the Los Angeles rattler results on identical materials and also with the toughness and Deval abrasion results on these same materials. The test results are given in the accompanying Table I and on the basis of the values obtained in the respective tests, ratings have been given to indicate the degree of excellence of the several rocks on the basis of the particular test used. A rating value of 1 indicates the best material tested and 9 the least desirable material in the series. In the Los Angeles rattler test the three low values are very close together, so close in fact that other tests made on the same samples might result in a reversal of their order of excellence. Bearing this in mind and comparing the ratings of the materials in the Los Angeles rattler test and in the roller test, it will be seen that there is an exceedingly close coincidence.

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[1] Director, City of Los Angeles Bureau of Standards, Los Angeles, Calif.
[2] Director, Bureau of Engineering, National Crushed Stone Assn., Washington, D. C.

TABLE I.—RESULTS OF VARIOUS TYPES OF TEST ON SOME TYPICAL AGGREGATES.

	Sample	Los Angeles Rattler		Roller Test	Toughness Test		Deval Test	
		Per cent Wear	Relative Rating	Relative Rating	Lowest Value	Relative Rating	Per cent Wear	Relative Rating
Granite	No. 1	28.9	4	4	8	6	2.6	6
Granite	No. 2	36.9	7	6	15	2	1.8	3
Greenstone	No. 3	23.2	2	3	12	3	1.7	1 or 2
Limestone	No. 4	34.9	5	5	5	8	3.5	8
Granite	No. 5	40.9	8	8	10	4	1.9	4 or 5
Granite	No. 6	21.2	1	2	24	1	1.7	1 or 2
Limestone	No. 7	23.4	3	1	7	7	2.9	7
Granite	No. 8	41.6	9	9	9	5	1.9	4 or 5
Slag	No. 9	36.2	6	7	..	..	..	......

It will also be noted that the ratings resulting from the toughness test do not compare at all well with those given by either the Los Angeles rattler or the roller tests and the same statement may be made with regard to the Deval abrasion test. In view of the fact that the roller test is really a service test, it is clear that neither the toughness test nor the Deval abrasion test can be relied upon to indicate service behavior, but, on the other hand, the Los Angeles rattler test does agree exceptionally well with service behavior. This conclusion is directly in line with the results obtained by the authors.

No doubt, as time goes on, additional information may show the desirability of assigning different test values to different materials because of their difference in angularity, but this will probably be slight and, accordingly, in the Los Angeles rattler we at last have an abrasion test which may be used in like manner on all three types of coarse aggregate with no necessity for making a change in the method of test because of the difference in characteristics of the fragments.

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[1] A. T. Goldbeck, J. E. Gray and L. L. Ludlow, Jr., "A Laboratory Service Test for Pavement Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 608 (1934).

The authors are to be congratulated on the excellence of their paper and for the extremely practical value of the results they have obtained. It is to be hoped that there will be no delay by the Society in adopting the Los Angeles rattler test as a standard method of testing aggregates.

A WEAR TEST FOR FLOORING MATERIALS

By J. R. Shank[1]

Introduction

The need for a satisfactory wear test for flooring materials is apparent to anyone who may have noticed the excessive wear that can take place at points on stairs and corridor floors and at other places where the traffic is particularly heavy. The stair tread at the newel post or the one place before the elevator push button where everyone places his foot are examples of such points. Here the stair or corridor traffic count figure must be multiplied many times. Floors patterned in beautiful designs using the many delightful effects that marbles can give may lose considerably when the observer finds that the whites are as much as $\frac{1}{8}$ in. lower than the blacks or greens. The architect may find himself in a difficult position when he must decide whether or not a new manufactured tile which may cost less might not well be substituted. Many times the wary architect will have nothing to do with any such new product because of his uncertainty, which retards progress in better building design and imposes an unfair load on the enterprising manufacturer. This paper has to do with a series of tests which indicate that an old apparatus now found in laboratories all over the country may satisfactorily be adapted to this test service.

A new apparatus and method of test for this service has been worked out by D. W. Kessler of the National Bureau of Standards[2] which operates on a square cross-section using No. 60 artificial corundum. The index is known as the Ha value and is worked out from a formula. A sand-blast apparatus is also on the market for this service.

Method of Test

Stones for road building have been tested in the past and to some extent are now being tested by means of a machine known as the Dorry apparatus.[3] Cylindrical cores are cut from a sample and submitted to a given amount of abrasion by silica sand, carried on cast steel. A test similar to this was exhibited in 1878 at the World's Fair at Paris. Professor

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[1] Research Professor, Engineering Experiment Station, Ohio State University, Columbus, Ohio.

[2] D. W. Kessler, "The Development of an Apparatus for Wear Tests on Flooring Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 855 (1928); "Wear Resistance of Natural Stone Flooring," National Bureau of Standards *Journal of Research*, Vol. II, No. 5, November, 1933, p. 635.

[3] L. W. Page, "Relations Between the Tests for the Wearing Qualities of Road-Building Rocks," *Proceedings*, Am. Soc. Testing Mats., Vol. XIII, p. 983 (1913); A. T. Goldbeck and F. H. Jackson, Jr., "The Physical Testing of Rock for Road Building," U. S. Department of Agriculture, Office of Public Roads, *Bulletin 44*, p. 7, June 10, 1912.

Johann Bauschinger of Munich made rather extensive tests by means of an apparatus of this nature and reported them in his Communications, Vol. XI, 1884.

The Dorry road-stone test must be modified somewhat to suit flooring materials. Floor units in service are set and fixed in position so that the wear comes on one face only. There is therefore nothing to be gained by applying the test to both sides and in different directions of the grain. The standard test for road stone is too severe; it cuts too deeply into the flooring specimen. The test for flooring materials should not be carried to depths greater than what may reasonably be expected for floorings as determined from practice. An index in terms of depth of wear appears to be more easily visualized and is more indicative than the standard Dorry coefficient expressed as a function of weight, particularly when the differences in specific gravity in floorings is considered.

The Dorry test is modified in the following ways so as to be better suited to flooring materials, particularly those of an earthy and generally inorganic type: Two similar cores are cut and fixed in the chucks, wearing face down and given 333 revolutions using standard Ottawa sand passing a No. 20 sieve and retained on a No. 30 sieve. The loss in grams is determined for the two and averaged. The apparent specific gravity is determined from one or two of the cores and the grams lost is translated over into cubic centimeters lost. This volume lost is divided by the cross-section of the core, giving the depth in millimeters worn off. This value of millimeters worn off is given as the wear coefficient. The results of this test may be expressed in the terms of the Dorry road-stone test by subtracting the grams lost from twenty.

Test Results:

The test was applied to a number of different kinds of possible flooring materials both natural and manufactured. This was possible because of the spirit of cooperation that was general among producers who generously sent in samples at their own expense. In all, about 220 samples were sent in and tested. The object of these tests is not to set before the reader the special properties of the various samples, but to furnish a background for the test so that the architect or other buyer, as well as the producer, may have a basis for judgment of tests that he may make or have made. It is also hoped that this brief start may be of service to the writer of specifications to furnish a basis of comparison for his own experience. No attempt was made to relate the results quantitatively with service conditions. It is believed that the judgment of an experienced user or reputable producer is of more value than any experimental connection studies made in a laboratory because of the wide variations in wearing conditions that must obtain in different geographical locations.

In the sample designations of Table I, the numerical portion indicates the supplier and the capital letter the several samples sent in by him. The small letters indicate cores cut different ways of the grain, "a" along, and "b" across the grain.

The depth wear coefficients, individually for samples which were used to make up Table I, are plotted graphically in Fig. 1. The graphs are plotted down from a base line to represent more nearly the normal wear action. It is of interest to note the wide variations found in each group. It cannot be said that a granite must necessarily be more resistant to wear than a marble or that quarry tile is more resistant than flagstone. The greatest resistance to wear is to be found among the porcelain tiles. It is worth noting that the black, green, and red marbles are all at the hard end of the marble group. The three sandstones and the gray-veined Vermont marble noted in Table I to be unsuitable for floor service are not included in Fig. 1 nor are the miscellaneous materials.

DETAILS OF TEST PROCEDURE

When cutting cores from all sorts of possible earthy, inorganic materials it was found that the diamond drill as usually found in laboratories was not satisfactory for cutting the tougher, harder, and denser materials such as porcelains and some of the granites. It was found to be unsuitable also for cutting neat cores from soft marbles under 1 in. in thickness. In the first case the action was much too slow, and in the second the pressure broke out the core when there was yet as much as $\frac{1}{4}$ in. to go. A piece of copper pipe fitted onto the drill press in place of the diamond drill bit with a small amount of No. 50 carborundum and water served to cut cores much more rapidly in the cases of the harder materials and more neatly for the softer ones. It was found that glass could be cut at the rate of 3 in. per hr. and it was possible to cut sandstones and marbles as fast as 10 or 12 in. per hr. Enough of the carborundum embedded itself in the copper to make the starting of a cut very easy. The carborundum was used over and over again by reintroducing the cuttings and the abrasive to the kerf by means of a small brush. It was necessary to lift the copper pipe bit periodically to redraw abrasive under the cutting edge. This required the close attention of an operator. It would not be difficult to make up automatic water and abrasive feed and bit lifting devices for a drill to be used extensively. It was found that the cores could be squared on the ends and finished on the cylindrical faces by means of an ordinary bench grinder. Care was taken to start the cut of the core from the face to be tested so that squaring up and finishing would be applied to that part to be held in the chuck.

Silica sand was used as an abrasive because it is believed that a very large part of the natural wear of floors is due to silica or softer material

TABLE I.—RESULTS OF WEAR TESTS.

Sample[a]	Source	General Description	Cores Tested	Apparent Specific Gravity	Wear Coefficient		Remarks
					Volume, cc.	Depth, mm.	
		SANDSTONES					
No. 1-A	Southern Ohio	buff and brown streaked	2	2.25	14.50	29.6	Not used for flooring
No. 1-B	Southern Ohio	pink mottled	2	2.31	12.45	25.4	
No. 1-C & D	Southern Ohio	buff and blue	4	2.15	5.80	11.86	
No. 6-A	Tennessee	pink-buff	2	2.53	0.47	0.96	
No. 6-B	New York	green-gray	2	2.56	1.43	2.91	
No. 6-C & D & 18-A	Tennessee	buff	6	2.53	0.93	1.90	
No. 34-A-G	New York	blue-gray	24	2.52	2.23	4.54[b]	
No. 45-A	New York	Medina stone-gray	4	2.67	0.64	1.31	
No. 45-B	New York	Medina stone-red	4	2.50	0.68	1.39	Paving block
No. 45-C & D	New York	gray	8	2.45	1.25	2.54	
No. 47-A & C	Northern Ohio	gray	8	2.15	10.23	20.83	
No. 48-A	Massachusetts	brown	2	2.16	6.88	14.02	
No. IX	Southern Ohio	blue-gray	2	2.26	2.14	4.36	
No. XVI	Northern Ohio	brown	2	2.09	12.82	26.1	Impregnated
No. O-3 & 4	Southern Ohio	blue-gray	8	2.22	3.22	6.55	Not used for flooring
		MARBLES					
[c]	Tennessee	pink	20	2.71	1.63	3.32	
No. 15-A-D	Georgia	pink and gray	6	2.72	3.41	6.95	
No. 25-A	Vermont	white	4	2.69	4.38	8.92	
No. 25-B	Vermont	black	2	2.72	1.18	2.40	
No. 25-C	Vermont	white	2	2.68	1.54	3.14	
No. 25-E	Vermont	garnet red	2	2.82	0.56	1.14	
No. 25-F	Vermont	light gray	2	2.68	2.48	5.06	
No. 25-G	Colorado	white-buff streaked	2	2.67	2.82	5.75	
No. 25-H	Vermont	green	2	2.63	0.17	0.35	
No. 25-I	Vermont	white-gray streaked	2	2.68	2.92	5.95	
No. 25-J	Vermont	white-gray mottled	2	2.69	2.83	5.77	
No. 25-K	Vermont	white	2	2.69	2.48	5.05	
No. 25-M	Tennessee	gray	2	2.70	1.96	3.99	
No. 25-N	Vermont	gray veined	2	2.71	4.50	9.16	Not used for flooring
No. 25-P	Vermont	gray, white streaked	2	2.71	2.70	5.50	
No. 25-Q	Vermont	gray	2	2.71	1.78	3.62	
No. 25-R	Alaska	gray, white lined	2	2.72	2.60	5.30	
No. 25-S	Vermont	gray	2	2.71	3.00	6.11	
No. 25-T	Vermont	white, heavy gray streaked	2	2.72	1.60	3.26	
No. 25-U	Vermont	light green	2	2.63	0.23	0.47	
No. 31-F	European	black	2	2.70	0.72	1.47	
No. 31-H	Italy	red	2	2.68	1.73	3.52	
No. 31-I	Italy	white	2	2.68	1.54	3.14	
No. 38	Italy	white Carrara	2	2.71	2.07	4.22	
No. 39	Unknown	apple, green onyx marble	2	2.76	1.65	3.36	An old scrap
No. 40-A	Minnesota	buff sandy texture	2	2.59	2.40	4.89	
No. 40-B	Minnesota	pink sandy texture	2	2.58	1.95	3.97	
No. 41-A	Missouri	buff veined	2	2.69	1.51	3.08	
		MARBLES—(*Continued*)					
No. 41-B	Missouri	pink	2	2.69	2.03	4.14	
No. 43-A	Missouri	buff	6	2.68	1.82	3.71	
No. 43-D	Missouri	buff-gray	2	2.63	2.57	5.23	
No. 43-E	Missouri	gray	4	2.64	2.37	4.83	
No. 44-B	Tennessee	pearl-gray	2	2.70	1.64	3.34	
No. 44-C	Tennessee	blue, veined	2	2.70	1.93	3.93	
No. 55-A	Alabama	brown mottled	2	2.69	1.11	2.26	
No. 57-E	Tennessee	red-brown mottled	2	2.71	2.35	4.78	
No. 57-G	Tennessee	dark red-brown	2	2.72	1.06	2.16	
No. X	Missouri	gray	2	2.66	2.15	4.38	
		GRANITES					
No. 3-A & 9-A	Wisconsin	black	4	2.89	0.41	0.84	
No. 3-B	Vermont	dark gray	2	2.64	0.47	0.96	
No. 4-A	Vermont	light gray	2	2.62	0.82	1.67	
No. 11-A & B	N. Carolina	light gray	4	2.61	0.36	0.73	
No. 13-A & B	Maine and New Hampshire	pink and green	4	2.60	0.82	1.67	
No. 13-C	Maine and New Hampshire	white	2	2.61	1.69	3.44	
No. 19-A	Maine	multicolored, large grained	2	2.60	1.14	2.32	
No. 46-A & C	N. Carolina	pink gray	8	2.61	0.30	0.61	Scrap paving block
No. 46-B	Unknown	gray	4	2.62	0.20	0.41	
No. 50-A	Minnesota	pink	2	2.69	0.48	0.98	
No. 50-B	Minnesota	variegated	2	2.65	0.54	1.10	
No. 50-C	Minnesota	gray	2	2.65	0.39	0.79	
No. 51-A	Texas	pink, very large grains	4	2.63	0.40	0.81	
No. 52-A	Maine	multicolored, large grained	4	2.63	0.365	0.74	
No. 53-A & 56-A	Massachusetts	pink	6	2.63	0.37	0.76	
No. 53-B	Massachusetts	gray	4	2.63	0.29	0.59	
No. 54-A	New Hampshire	pink	6	2.61	0.42	0.85	
No. 54-B	New Hampshire	buff gray	6	2.63	0.52	1.06	
No. III	New Hampshire	gray, fine grained	2	2.63	0.60	1.22	
		LIMESTONES, TRAVERTINES, ETC.					
No. 10-A	Indiana	gray-colitic, coarse grained	2	2.34	3.34	6.80	
No. 10-B	Indiana	medium grained, with holes	2	2.44	2.38	4.85	
No. 10-C	Indiana	fine grained	2	2.29	3.45	7.03	
No. 16-A	Indiana	gray, fine grained	2	2.32	4.85	9.88	
No. 16-B	Indiana	buff, coarse grained	2	2.30	3.28	6.69	
No. 16-C	Indiana	buff, fine grained	2	2.32	6.15	12.52	
No. 17-A	Germany	bright buff	4	2.59	0.77	1.57	
No. 25-D	Italy	travertine	2	2.49	2.20	4.48	
No. 59-A	Ohio	Columbus formation	4	2.57	2.62	5.33	
No. VI	Indiana	St. Paul, dense white	2	2.68	1.48	3.02	
No. XII	United States	travertine	2	2.54	2.44	4.97	
No. XV	Indiana	buff-colitic, medium grained	2	2.29	6.91	14.08	

SLATES

No. 23-A

SLATES

No. 23-A	Pennsylvania	blue	2	2.76	3.31	6.74	
No. 35-Aa	Vermont	red	2	2.79	2.27	4.62	
No. 35-Ba	Vermont	unfading green	2	2.78	1.71	3.48	
No. 35-Bb	Vermont	unfading green	2	2.78	1.20	2.44	
No. 35-Ca	Vermont	green and purple	2	2.80	1.97	4.01	
No. 35-Cb	Vermont	green and purple	2	2.79	1.18	2.40	
No. 35-Da	Vermont	mottled gray	2	2.78	2.17	4.42	
No. 35-Ea	Vermont	purple	2	2.80	1.74	3.54	
No. 35-Eb	Vermont	purple	2	2.81	0.89	1.81	
No. 35-Fa	Vermont	green	2	2.78	2.47	5.03	
No. 35-Ga	Vermont	unfading green	2	2.79	2.20	4.48	
No. 35-Gb	Vermont	unfading green	2	2.79	1.25	2.54	
No. XIV	Pennsylvania	blue	2	2.74	5.00	10.20	Switch board

CEMENT PRODUCTS

No. 7-A	Imitation granite, aluminous grain	2	2.32	0.46	0.94	
No. 7-B	Imitation Botticino, aluminous grain	2	2.20	2.30	4.69	
No. 7-C	Imitation marble, aluminous grain	2	2.28	0.99	2.02	
No. 7-D	Imitation granite, alundum aggregate	2	2.22	0.53	1.08	
No. 7-E	Imitation Botticino, alundum aggregate	2	2.23	1.10	2.24	
No. 7-F	Imitation marble, alundum aggregate	2	2.10	2.42	4.93	
No. 7-G	Imitation granite, no abrasive	2	2.28	1.26	2.57	
No. 7-H	Imitation marble, no abrasive	2	2.19	2.16	4.40	
No. 8-A	Imitation marble, non-slip	2	2.18	2.73	5.66	
No. 8-B	Imitation marble, marble aggregate	2	2.26	2.12	4.32	
No. 8-C	Imitation granite, granite aggregate	2	2.20	1.67	3.40	
No. 30-A	terrazzo, black and white aggregate	2	2.19	2.63	5.36	Light-weight aggregates were used for all specimens marked No. 30.
No. 30-B	terrazzo, multicolored gray	2	1.66	2.83	5.77	
No. 30-C	terrazzo, buff colored	2	2.07	2.21	4.50	
No. 30-D	terrazzo, buff aggregate	2	2.10	2.15	4.38	
No. 30-E	terrazzo, gray aggregate	2	1.70	2.51	5.12	
No. 30-F	terrazzo, yellow and brown aggregate	2	2.11	3.43	6.99	
No. 30-G	terrazzo, multicolored aggregate	2	2.30	1.57	3.20	
No. 30-H	terrazzo, multicolored aggregate	2	2.12	2.10	4.28	
No. 30-I	terrazzo, black, pink and white aggregate	2	2.31	3.27	6.66	
No. 30-J	terrazzo, black, pink and yellow aggregate	2	2.12	2.15	4.38	
No. 30-K	terrazzo, yellow cement	2	2.09	2.14	4.36	
No. 30-L	terrazzo, white and black aggregate	2	1.78	3.76	7.66	
No. 30-M	terrazzo, gray dull effect	2	1.52	3.66	7.45	
No. 31-A	terrazzo, green-black	2	2.36	1.37	2.79	
No. 31-B	terrazzo, garnet and white aggregate	2	2.44	2.26	4.61	
No. 31-C	terrazzo, buff colored	2	2.44	1.83	3.73	
No. 31-D	terrazzo, buff and white aggregate	2	2.43	1.80	3.67	
No. 31-E	terrazzo, white aggregate	2	2.32	3.30	6.72	
No. 49-A-B	surface treated cement product	4	2.31	0.79	1.62	
No. VII	terrazzo, white aggregate	2	2.36	1.57	3.20	
No. VIII	cut cast stone	2	2.42	1.92	3.91	

CERAMIC PRODUCTS

No. 12-A	white 4 by 4 in. glazed porcelain	2	2.23	0.39	0.80	
No. 12-B	white 4 by 4 in. glazed porcelain	2	2.26	0.38	0.78	
No. 12-C	white 4 by 4 in. porcelain unglazed	2	2.38	0.29	0.59	

CERAMIC PRODUCTS—(*Continued*)

No. 12-E	white 4 by 4 in. porcelain unglazed	2	2.37	0.34	0.69	
No. 12-F	white 1½ by 1½ in. porcelain mosaic tile	2	2.20	0.24	0.49	
No. 12-G	white 2¼ by 2¼ in. porcelain mosaic tile	2	2.35	0.13	0.26	
No. 14-N	light buff granular 4 by 4 in. mosaic tile	2	2.00	0.82	1.67	
No. 14-O	buff 4 by 4 in. granular tile	2	2.03	0.86	1.75	
No. 14-P	buff 4 by 4 in. granular tile	2	2.02	1.09	2.22	
No. 17-B	large brown Welsh quarry tile	2	2.29	0.46	0.94	
No. 17-X	large brown Welsh quarry tile	2	2.33	0.18	0.37	
No. 20-A	red brown quarry tile	2	2.26	1.00	2.04	
No. 20-B	brown quarry tile	2	2.25	0.83	1.69	
No. 20-C	brown quarry tile	2	2.23	1.25	2.55	
No. 21-A	light red quarry tile	2	2.25	1.08	2.20	
No. 21-B	red quarry tile	2	2.21	0.64	1.30	
No. 21-C	red quarry tile	2	2.23	1.26	2.57	
No. 22-A	special shape red quarry tile	2	2.47	0.58	1.18	
No. 22-B	red quarry tile	2	2.45	0.16	0.33	
No. 22-C	red-brown quarry tile	2	2.17	4.50	9.17	
No. 28-A	brown quarry tile	2	2.10	3.25	6.62	
No. 28-B	gray quarry tile	2	2.09	3.30	6.73	
No. 28-C	red quarry tile	2	2.14	2.15	4.38	
No. 28-D	red-brown quarry tile	2	2.08	3.28	6.69	
No. 29-A	small dark red tile	2	2.20	2.00	4.08	
No. 29-B	small dark red tile	2	2.20	3.30	6.73	
No. 29-C	red quarry tile	2	2.41	0.93	1.90	
No. 29-D	red quarry tile	2	2.43	0.55	1.12	
No. 29-E	red quarry tile	2	2.42	0.52	1.06	
No. 29-F	dark-red special shape tile	2	2.25	1.34	2.73	
No. 33-A	brown tile	2	2.34	0.68	1.38	
No. 33-B	white porcelain tile	2	2.40	0.08	0.16	
No. 33-C	green porcelain tile	2	2.44	0.07	0.14	
No. 33-D	black tile	2	2.50	0.44	0.90	
No. 36-A	special stair-tread block	2	2.29	1.27	2.59	
No. 36-B	special stair-tread block	2	2.30	1.17	2.38	
No. 36-C	special stair-tread block	2	2.29	1.21	2.46	
No. 36-D	special stair-tread block	2	2.29	1.21	2.46	
No. 37-A	de-aired sewer pipe wall	2	2.43	0.18	0.37	37-A & 37-B from same body
No. 37-B	sewer pipe wall not de-aired	2	2.35	0.45	0.92	
No. 37-C	flat, top liner plate	2	2.21	1.46	2.98	
No. 37-D	flat, bottom liner plate	2	2.23	1.77	3.61	
No. PBA	de-aired paving brick	2	2.40	0.25	0.51	
No. PBB	de-aired paving brick	2	2.40	0.27	0.55	
No. I	small blue hexagonal mosaic tile	2	2.36	0.16	0.33	
No. IV	ordinary paving brick	2	2.35	0.41	0.84	
No. V	buff quarry tile	2	2.26	0.60	1.22	
No. XI	red quarry tile	2	2.16	2.42	4.93	

MISCELLANEOUS

No. 5-A	composition stone	2	2.84	1.44	2.93
No. 26-A	magnesite-wood chips composition	4	1.06	2.34	4.72
No. 32-A	abrasive material composition	2	2.89	0.81	1.65
No. 32-B	abrasive set in rubber	2	1.51	0.07	0.14
No. 47-B	artificial sandstone	2	1.92	5.21	10.61
No. 58-A	special pressed fiber board	2	1.09	1.09	2.22
No. II	white opaque glass	2	2.48	0.48	0.98

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[a] The numbers indicate the supplier, the capital letters the several samples sent by him, "a" the cores cut along the grain, and "b" the cores cut across the grain.

[b] New York sandstones, 34 range from 1.22 to 12.42 mm. worn off.

[c] This embraces 10 different marble samples ranging from 2.62 to 3.93 mm. worn off.

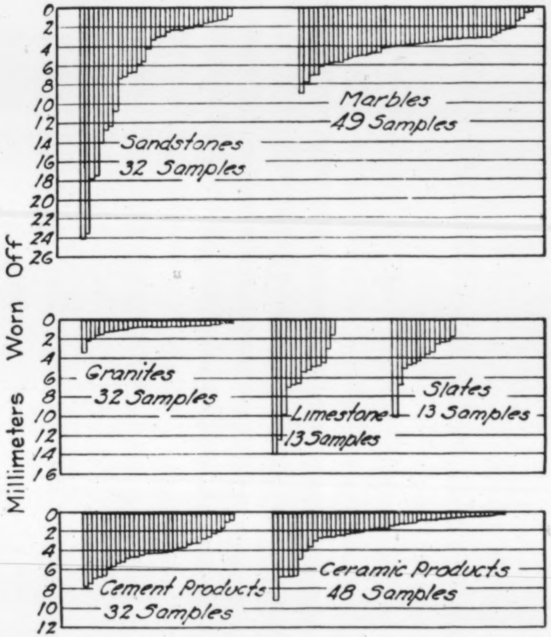

FIG. 1.—Data of Table I Shown in Order.

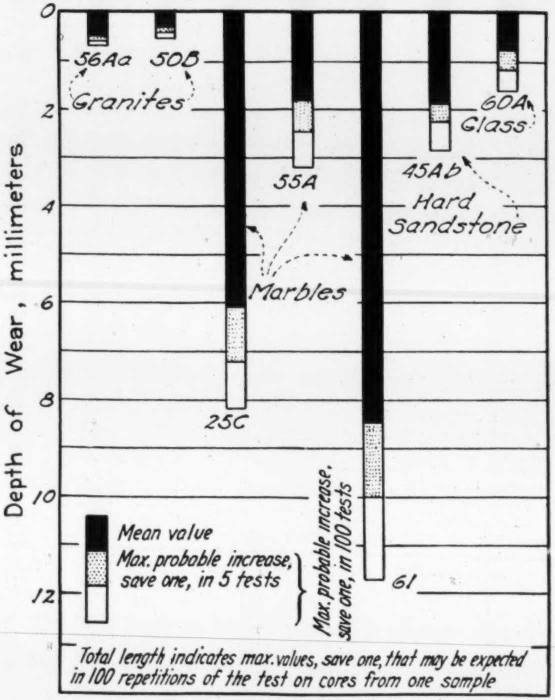

FIG. 2.—Probable Variations. Data from Table II.

carried on the shoe soles. The probability of harder material being present is very small. Standard Ottawa silica sand passing a No. 20 sieve and retained on a No. 30 sieve, as used for cement testing, was used because it is very likely to be found in all testing laboratories and because it is dependably uniform. The difference between No. 30 - No. 40 and No. 20 - No. 30 white silica sand is so slight that the presence of both of them in one laboratory is likely to be the source of errors and confusion. The larger sand, so long as it is kept clean of dirt and broken particles of material, seems to give more uniform action under the test core. A single layer of the sand is dragged under the core. If this layer is lost because of no feed or the sticking of the chuck in the guide, the change in sound is at once apparent and the fault may be remedied. It was found that the sand should be cleaned of both dust and larger particles by screening as often as every ten test runs. Totally new sand should be run under old or scrap cores for one or two runs before test cores are introduced.

No account was taken of the weight of the cores. Cores may be expected to weigh not over 50 g. This amounts to an excess of only 4 per cent over the 1250-g. weight of the chuck. As will be shown later, variations in the wearing resistance of different layers of the same core is very likely to be more than 4 per cent.

The apparent specific gravity was found by weighing the cores in and out of water. The more porous materials were given a coat of orange shellac after the dry weight was taken, which was allowed to dry before making the immersed weight determination. The less porous ones such as most of the marbles were soaked and mopped dry.

STUDIES ON PROBABLE ERRORS AND VARIATIONS

Before a test like this can be employed in specifications some knowledge must be had as to what variations or errors must be expected for the test and what variations must be expected for the material. Breadths of tolerance must be expressed or be implied in the specifications to avoid hardship to the producer and consequently high cost to the purchaser.

In order to obtain and present values for the probable error of the test a number of samples were given many repetitions of the test. In some cases where the sample was quite hard, the repetitions were all made on one pair of cores. In others, a large number of cores were cut from the sample and only one or two runs were made on any one pair of cores. Two granites three marbles, one sandstone, and one glass were selected for this work. One granite, No. 56-Aa, was from the eastern part of the country and the other, No. 50-B, was from the middle west. Both were pink and coarse-grained. One marble, No. 25-C, from Vermont was nearly white and quite uniform in texture, and another, No. 55-A, from Alabama was brown, variegated, and non-uniform in texture. The third marble, No. 61, was unknown, possibly Italian Carrara. The sandstone, No. 45-Ab, was a Medina sandstone paving block, quite uniform in its texture for sandstone. The glass, No. 60-A, was a piece of opaque white glass which was selected to fill the place of a dependably uniform material for calibrating the test but did not serve the purpose. Worn surfaces at different depths showed markedly different textures; at one time coarse and at another fine. The results of these tests are shown on Table II.

The probable error values shown are all computed by the method of least squares. One unit for computations was the average of a pair of cores or the result of one test run. The difference shown in some cases between the number of tests run and the number of tests used in the calculations came about because in some cases certain of the tests were judged to be wild. By the theory of probability, the limiting error value for all but one error in a given number of observations may be found as a coefficient times the probable error. In the reverse order this might be expressed as: the probability that an error *a* is greater than 3.43 times the probable error ***r*** is 1 to 50. If fifty observations are made, only one of them may have an error greater than 3.43***r***. If ten are made, the limit is 2.42***r*** or if five are made, the limit is 1.90***r***. If two are made, the limit is ***r***, which coincides with the definition of the probable error itself which is usually given as "That particular error which is just as likely to be exceeded as not,"[1] or in other words, one error is likely to be greater than the probable error and one is likely to be less, since the mean is the most probable value. The calculations for this coefficient are taken from "Adjustment of Observations" by Wright and Hayford.

TABLE II.—PROBABLE ERROR DETERMINATIONS.

	Pink Milford Granite No. 56-Aa	Cold Spring Granite No. 50-B	White Vermont Marble No. 25-C	Brown Alabama Marble No. 55-A	White Unknown Marble No. 61	Red Medina Sandstone No. 45-Ab	White Opaque Glass No. 60-A
Numbers of cores cut	2	2	99	20	43	4	5
Number of test runs	32	40	50	50	43	47	40
Average number of tests per core	32	40	1	5	2	23.5	16
Number of test runs in computations	32	39	50	50	42	47	37
Average, worn off, cu. cm	0.250	0.165	3.02	0.900	4.20	0.840	0.390
Average depth of wear, mm	0.510	0.34	6.15	1.83	8.55	1.71	0.795
Probable error or variation, per cent	10.6	17.1	9.45	20.0	9.9	18.4	27.1
Maximum variations, save one, for various numbers of tests: 1 in 5	20.2	32.5	18.0	38.0	18.8	34.9	51.6
1 in 10	25.8	41.4	22.9	48.4	24.0	44.0	65.7
1 in 100	40.8	65.5	36.1	76.5	38.0	69.7	104.0

An attempt was made to get a dependably uniform material such that a large number of test runs made from the same sample would indicate the probable error for the test alone. The best, as Table II will show, was the piece of white Vermont marble. The other piece of white marble was almost as good. The pink Milford granite was in this same class in spite of the large crystals in it. As it is rather difficult to say that any marble or granite is absolutely uniform, no statement can be made as to the probable error of the test alone. It is evidently less than 9.45 per cent, but how much less is not certain. For the sake of comparison, standard cement specifications will allow variations in three tension briquets of as much as 15 per cent when they are made, cured and tested under the same conditions.

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[1] I. S. Sokolnikoff and E. S. Sokolnikoff, "Higher Mathematics for Engineers and Physicists," McGraw-Hill Book Co., Inc., New York City (1934.)

The merits of the test may be summarized as follows:

1. It is similar to a well-known test and the results may be expressed in terms of that test so that direct comparisons can be made with past experience.

2. No new apparatus need be bought save for the substitution of a piece of copper pipe for the diamond drill bit on the old drill press. Any small drill press may be brought into service thus doing away with the diamond drill entirely.

3. The index is one easily visualized by both buyer and producer, and values can therefore more easily be kept in mind.

4. The abrasive used is of the same material as that which produces natural wear on floors.

5. The apparatus employed is now found in all well-equipped testing laboratories.

6. The probable error of the test is within the variation of the materials tested.

Acknowledgment.—The author wishes to acknowledge the assistance rendered by Messrs J. R. Katz and William Levy, students, Ohio State University.

DISCUSSION

MR. D. W. KESSLER.[1]—I believe the wear test developed by Mr. Shank (which can be readily applied by most commercial laboratories) is very commendable, and the data supplied should receive serious consideration among architects.

There are two or three inherent faults in the Dorry apparatus which I should like to point out. I think they can be readily overcome by mechanical changes. The first of these is the manner of holding the specimen on the grinding disk. It is held in a chuck which simply slides down through a sleeve as wear shortens the specimen. When the disk is revolved the lower end of the chuck is pressed against one side of the sleeve and the top is pressed against the other. It would seem that this would produce considerable constraint to the downward movement of the chuck during the grinding process, which would result in unequal pressures of the specimen on the disk during the test.

Another point that is undesirable is the fact that the small specimen is held in one position on the disk during the grinding operation, which usually results in grinding a rut in the disk. The Bureau of Public Roads has overcome this in their apparatus by imparting a to-and-fro motion to the specimen during the grinding process.

I believe both of these faults could be overcome by hinging the chuck on a vertical rod and also imparting the to-and-fro motion. The hinge would be free to move downward as the specimen is shortened. The sleeve would be eliminated and friction in the hinge reduced to a minimum by the to-and-fro motion.

There is one other point I should like to emphasize. A 1-in. core is rather small and probably does not give as representative a sample as a larger specimen. When you have only a small core it is necessary to grind to a considerable depth in order to get a measurable amount of wear. On some types of artificial flooring materials the wearing surface is very thin and unless the test is applied to a very thin film you have not measured the wear resistance that is desired. Also, one frequently wants to make a determination on the effectiveness of surface hardening treatments, particularly concrete hardeners. Some such treatments are quite effective in increasing the wear resistance on a very shallow layer. In order to determine the value of such treatments the test must be confined to a depth of less than 1 mm.

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[1] Chief, Stone Section, National Bureau of Standards, Washington, D. C.

MR. G. W. BAIN[1] (*presented in written form*).—The results obtained by Mr. Shank with the modified Dorry machine and by Mr. Kessler with his apparatus show essentially similar behavior of flooring materials under the conditions of the test. My experience with the marble group of flooring materials would indicate that both these tests rate the material in an order that is not their order of merit in actual installations. Mr. Kessler encountered similar irregularities in behavior between laboratory tests and service tests.

Vanderwilt, while studying polishing, found that a great difference existed between rolling abrasion and scratch abrasion. The abrasion reported by both Kessler and Shank is rolling abrasion and the marks produced on the affected surface are predominantly "chatter-marks." The marks on floors are rarely the chatter type; they are a series of parallel scratches and are the scratch abrasion of Vanderwilt. These parallel scratches are due to sharp particles becoming embedded partially in leather or rubber shoe soles and scratching the floor as the shoe moves over it; they roll only a short distance at the end of the shoe drag.

It is evident that any test which is going to rate materials for service conditions must simulate service conditions and accordingly test resistance to scratch abrasion. Actually many stones which are weak to rolling abrasion are strong to scratch abrasion and *vice versa*. I suggest substituting leather or rubber for the metal bed of the Dorry testing machine. I have been informed that Mr. Shank has made a number of tests on this modified bed and that the results give to flooring materials of the marble class a different order rating and one which is more accordant with experience in installations. I hope that Mr. Shank will favor us with these more recent results.

MR. M. O. WITHEY[2] (*presented in written form*).—From time to time during the past 15 yr. a modified form of the Dorry abrasion test has been used in the Materials Testing Laboratory at the University of Wisconsin. With this test determinations have been made of the resistance to attrition of plain mortars, mortars which have been treated with hardeners and also of specially prepared wood coverings for floors, on rubber soles for shoes, and on composition floor coverings similar to linoleum.

The types of spool and test specimen used for this work are shown in the accompanying Fig. 1. It will be observed that the surface of the specimen subjected to abrasion is considerably larger than that employed in the standardized Dorry test. This, we believe, is a decided advantage. In testing linoleum or other fabric, it has been customary to cement the material under test to a dummy similar to the mortar specimen shown in the illustration. In the tests on mortars and hardeners, the specimens were weighed after 500, 1000, and 2000 revolutions in order to determine the rate of wear at the surface and at a short distance from the surface. For tests on composition floor coverings the weight on each spool was increased to 23.5 kg.

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[1] Assistant Professor of Geology, Department of Geology, Amherst College, Amherst, Mass.
[2] Professor of Mechanics, University of Wisconsin, Madison, Wis.

This type of test is quite satisfactory for the mortars and for the composition floor covering. Wood suffers considerable wear under this test. The test did not prove effective in measuring the wear of rubber soles. In the latter tests, little if any wear was produced when the specimens were loaded with the 1100-g. weight, and if 50 lb. per spool were added, abrasion was accompanied by burning of the rubber.

MR. J. R. SHANK.[1]—The points brought out in discussion are generally well taken. The test as here described is definitely an accelerated test and, as such, is not free from the faults which such test are likely to have. Mr. Bain points out that the action is not similar to that of the shoe sole on the floor; that the test produces rolling abrasion rather than scratch abrasion. The observation made by the author is that it more nearly simulates the action of the jumper drill than that of the shoe sole. The hardness of the disk face, together with the mass of the disk, and the rounded, though not spherical sand particles produce a vibration on the specimen and chuck which produces this drill action. This also obviates some of the defects pointed out by Mr. Kessler. The vibration of the chuck in the guides keeps it always loose, and the motion of the disk under it causes a slow turning action on the chuck and specimen, so that the action is not all one way, and there are therefore no rut marks on the specimens.

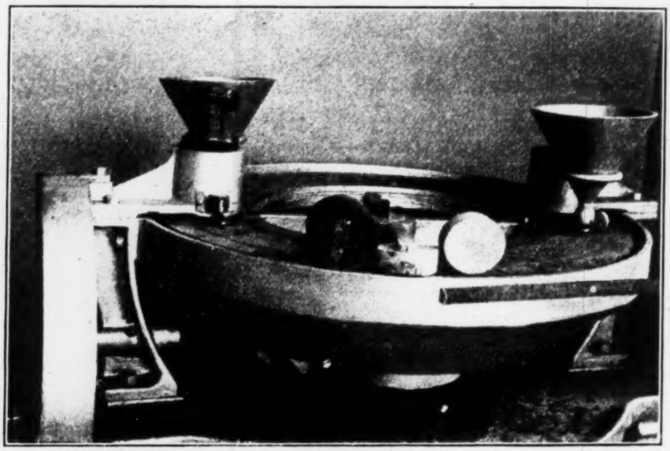

FIG. 1—Types of Spool and Specimen Used in the Abrasion Test.

The test best suits natural rocks and manufactured products which are uniform throughout, and is not well suited for testing products having special surfaces. It is also not well suited to testing surface textures, and is definitely not suitable for tough fibrous or grained materials such as linoleum, mastic, or wood floors.

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[1] Research Professor, Engineering Experiment Station, Ohio State University, Columbus, Ohio.

The modification in the apparatus spoken of by Mr. Bain, where rubber is used to face the cast steel disk, has been made and work is now going on. This is more nearly like the action of the shoe sole, and the drill action has disappeared. It was found that too little action was had from the rounded sand so that it became necessary to substitute crushed quartz of a definite size. It was also found necessary to prepare fresh crushed quartz by giving it a number of preparatory runs after which it could be used for only a very few test runs. The time necessary and the cost of this revised test will be greater. It will be better suited for testing special surfaces and textures.

A STUDY CONCERNING A MODIFICATION OF THE PRESENT MISCIBILITY TEST AND ITS RELATIONSHIP WITH PROBABLE SETTLEMENT RESULTS

By C. A. Downing[1]

Synopsis

This paper is based on utilization of the original conception of the miscibility test, that is, extreme dilution of mixing emulsions in the ratio of 50 ml. of emulsion to 150 ml. of distilled water serves as an accelerated stability test. Expensive delay and inconvenience is often experienced by both consumers and producers of asphalt emulsions on account of being forced to wait for the release of materials due to certain specified tests. A method has been devised that will predict for all practical purposes the probable 5-day settlement results.

The apparatus used is simple and inexpensive; and the method suggested is one that can be carried on in conjunction with present routine tests. It has the advantage that a complete release on the material in question may be made within one day after the sample is received in the testing laboratory.

The tabulated data cover a wide range of different type mixing emulsions produced by six different producers that merchandise emulsions on a national basis. The data cover materials produced in nine different plants and include emulsions that were prepared from both Mexican and domestic asphalts.

Introduction

The interpretation of the usual miscibility test on asphalt emulsions as outlined in the A.S.T.M. Tentative Methods of Test for Emulsified Asphalts (D 244 - 34 T)[2] have been a continual source of controversy in our laboratory. As an example, some emulsions have a tendency to stratify or form a different colored layer on dilution. This has been explained by some as an evidence of so-called "creaming" which was not considered detrimental. It has always been the author's interpretation of such a test that a perfectly stable emulsion should not stratify but should remain perfectly uniform in color when diluted with distilled water and permitted to stand for 2 hr. The question as to what constitutes an "appreciable" coagulation of the asphalt content of the emulsion, or as it was stated in former years "an appreciable separation," therefore presents an opportunity for a difference of opinion between the testing laboratory and some producers.

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[1] Engineer in Charge, Municipal Testing Laboratory, St. Louis, Mo.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 926 (1934); also 1934 Book of A.S.T.M. Tentative Standards, p. 711.

With this thought in mind, experimental work was started to evaluate, if possible, that quantity of asphalt which might be termed "appreciable." As the work progressed, it was noted that the maximum difference in the asphaltic contents of the top, middle and bottom levels of diluted emulsion had some relationship with the probable 5-day settlement results. This relationship was sufficiently close to permit the prediction as to whether or not the sample would comply with the specified 5-day settlement requirements. Since an earlier release of material by a laboratory was of much greater practical importance to both consumers and producers, the original intent was somewhat minimized although the results clearly differentiate between extremely stable emulsions and those not quite so stable.

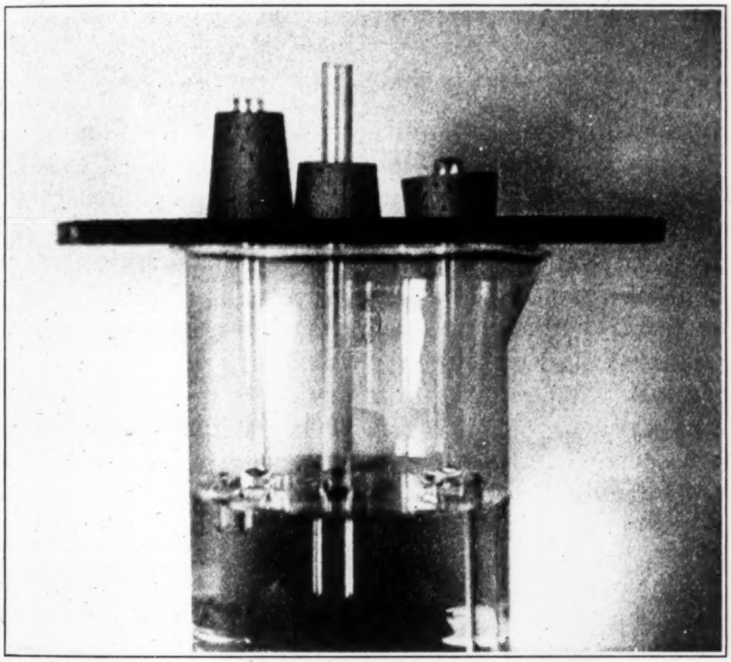

FIG. 1.—Apparatus for Modified Miscibility Test.

Test apparatus was developed and a method adopted which permits uniform testing of diluted emulsions after observations have been made as to whether they comply with the standard miscibility tests.

TESTING APPARATUS

The apparatus used in making this test consists of three 7-mm. glass tubes cut to convenient lengths. Bored corks are placed on the tubes so that when the tubes are used as pipettes to extract samples from the dilute emulsion in the 400-ml. beaker, their predetermined depth in the liquid will remain constant. A small piece of cigar-box wood 6 by 12 cm. in section with three 10-mm. holes in it is used to cover the beaker. This together with the corks on the tubes produces a stable base for uniform extraction of the samples from the top, middle and bottom levels of the liquid. In order to have a uniform depth of dilute emulsion, 400-ml. beakers have been selected as the standard containers for our miscibility tests. Figure 1 shows this apparatus in place on such a beaker containing 200 ml. of water. It will be noted that the lengths of the three tubes are such that samples can be taken from three different levels of the liquid. The extracted samples are evaporated in ordinary 15 to 25-ml. crucibles.

PROCEDURE

The standard miscibility test is made on the emulsion in the usual manner. At the conclusion of this test and after visual examination is made of the sample, the wood is placed on top of the beaker and the shortest glass tube is inserted through the wooden strip into the diluted emulsion. The tube is used as an ordinary pipette and the sample taken of the material at the top level is placed in a previously weighed crucible. It may be necessary to make two or three extractions to obtain a sample that will weigh approximately 1 g. The medium and long glass tubes are then used in sequence and samples taken from the middle and bottom levels. It is necessary to cover the end of the tube when first inserting it in the emulsion to prevent contamination or dilution of the sample when the tube passes through the different levels of emulsion. It is also advisable to clean the tubes on the outside, especially when extracting samples from the middle and bottom levels. This can be done easily by wiping the tube with a cloth before the volume contained in the tube is released into the crucibles. The three crucibles containing samples from the top, middle and bottom levels are then weighed and evaporated at 325 F. for a period of 2 hr. and are then permitted to cool and are reweighed and the asphaltic content determined. It will be found that there will be some variation in the asphaltic content in the three samples, and the lowest value is subtracted from the highest value found by this method. The maximum difference in these values will indicate the probable behavior of the emulsion when subjected to the settlement test.

DISCUSSION OF TEST DATA

Table I shows the results of settlement, miscibility and modified miscibility tests made on 69 different samples of mixing emulsions produced by six different emulsion companies at nine different plants.

It is evident from Table I that visual examination of the ordinary miscibility test is not sufficiently accurate to warrant acceptance or release

TABLE I.—RESULTS OF SETTLEMENT, MISCIBILITY AND MODIFIED MISCIBILITY TESTS.

Sample	A.S.T.M. 5-Day Settlement Test	A.S.T.M. Miscibility Test	Percentage of Asphalt Modified Miscibility Test			Maximun Difference
			Top	Middle	Bottom	
No. 1	0.0	OK	16.2	15.8	16.2	0.4
No. 2	0.0	OK	15.2	15.3	16.2	1.0
No. 3	0.1	OK	13.7	14.3	14.5	0.8
No. 4	0.1	OK	13.7	13.4	14.4	1.0
No. 5	0.1	OK	11.2	11.0	12.4	1.4
No. 6	0.15	OK	18.3	21.2	17.9	3.3
No. 7	0.2	OK	14.3	14.9	15.1	0.8
No. 8	0.2	OK	21.2	20.7	17.1	4.1
No. 9	0.3	OK	13.2	13.5	13.5	0.3
No. 10	0.3	OK	16.7	15.7	15.7	1.0
No. 11	0.3	OK	18.0	19.0	17.8	1.2
No. 12	0.4	OK	15.1	14.9	15.4	0.5
No. 13	0.4	OK	14.7	14.5	15.1	0.6
No. 14	0.5	OK	11.9	12.3	12.6	0.7
No. 15	0.5	OK	15.8	16.5	16.8	1.0
No. 16	0.6	OK	13.1	13.8	13.8	0.7
No. 17	0.6	OK	13.5	13.8	15.1	1.6
No. 18	0.7	OK	16.7	16.6	17.1	0.5
No. 19	0.7	OK	14.2	14.7	15.7	1.5
No. 20	0.8	OK	14.0	14.3	15.1	1.1
No. 21	0.8	OK	15.1	15.8	16.4	1.3
No. 22	0.9	OK	12.8	14.2	15.0	2.2
No. 23	1.0	OK	15.3	15.2	15.2	0.1
No. 24	1.0	OK	12.3	12.6	12.9	0.6
No. 25	1.0	OK	10.6	11.8	13.2	2.6
No. 26	1.0	OK	16.4	16.6	19.1	2.7
No. 27	1.1	OK	17.2	16.3	17.1	0.9
No. 28	1.2	OK	12.9	13.4	14.0	1.1
No. 29	1.2	OK	12.6	13.3	14.0	1.4
No. 30	1.3	OK	14.6	13.5	14.9	1.4
No. 31	1.3	OK	15.4	16.8	17.9	2.5
No. 32	1.6	OK	16.8	16.7	17.0	0.3
No. 33	1.7	OK	12.7	13.1	14.0	1.3
No. 34	1.8	OK	14.1	13.9	14.2	0.3
No. 35	1.8	OK	15.1	15.2	14.7	0.5
No. 36	1.9	OK	17.1	16.7	16.9	0.4
No. 37	1.9	OK	13.8	12.4	13.6	1.4
No. 38	2.1	OK	12.0	12.7	13.1	1.1
No. 39	2.1	OK	18.1	17.1	16.6	1.5
No. 40	2.1	OK	9.0	12.8	13.1	4.1
No. 41	2.3	OK	17.5	18.3	20.6	3.1
No. 42	2.4	OK	14.8	16.1	18.6	3.6
No. 43	2.8	OK	14.5	14.7	15.1	0.6
No. 44	3.1	OK	14.7	14.7	15.7	1.0
No. 45	3.4	OK	14.7	12.4	15.5	3.1
No. 46	3.7	OK	15.0	15.0	14.6	0.4
No. 47	4.0	OK	15.5	14.7	17.2	2.5
No. 48	5.6[a]	OK	19.0	20.5	25.0	6.0
No. 49	6.1[a]	OK	15.7	17.9	22.0	6.3
No. 50	6.7[a]	NG[a]	19.8	17.5	14.3	5.5
No. 51	6.8[a]	OK	15.4	13.7	18.2	4.5
No. 52	7.0[a]	OK	13.8	16.9	20.0	6.2
No. 53	7.8[a]	NG[a]	10.4	9.8	39.1	29.3
No. 54	8.45[a]	OK	13.3	13.1	30.0	16.9
No. 55	8.75[a]	NG[a]	10.6	10.5	40.0	29.5
No. 56	9.6[a]	OK	14.0	13.0	19.5	6.5
No. 57	10.6[a]	OK	12.5	13.3	18.7	6.2
No. 58	10.7[a]	OK	15.8	17.9	27.1	11.3
No. 59	10.85[a]	NG[a]	1.7	7.2	34.5	32.8
No. 60	14.0[a]	OK	15.4	16.3	21.3	5.9
No. 61	14.45[a]	NG[a]	12.8	13.3	17.5	4.7
No. 62	15.6[a]	OK	11.5	16.2	29.0	17.5
No. 63	18.2[a]	OK	7.5	10.6	35.8	28.3
No. 64	19.6[a]	NG[a]	4.1	21.9	34.5	30.4
No. 65	20.35[a]	OK	11.9	12.5	27.4	15.5
No. 66	21.7[a]	OK	13.1	13.9	33.4	20.3
No. 67	28.0[a]	OK	9.6	13.6	34.9	25.3
No. 68	31.05[a]	NG[a]	6.7	7.0	39.9	33.2
No. 69	40.3[a]	NG[a]	5.6	6.1	37.7	32.1

[a] Indicates deviation from A.S.T.M. Tentative Specifications for Emulsified Asphalt (for Coarse Aggregate Plant Mixes) (D 397 - 34 T), and for Emulsified Asphalt (for Retread and Coarse Aggregate Mixes) (D 398 - 34 T), (*Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, pp. 916 and 918, respectively (1934); also 1934 Book of A.S.T.M. Tentative Standards, pp. 684 and 686, respectively), 5 being maximum settlement permitted under requirements for mixing emulsions.

of the material. Many of the results shown indicate that the miscibility tests were perfectly satisfactory but the 5-day settlement tests were in excess of the specified limits of five.

In any discussion of asphalt emulsions, the true nature of such materials and their method of use in the construction field must be borne in mind. It is generally considered that an asphaltic emulsion is an aqueous liquid in which are dispersed minute particles of asphalt. The nature of the material is such that it can be either sprayed or mixed by mechanical means with aggregate so as to form a thin coating on such aggregate. In many instances, it is quite common to use emulsions with aggregates that are wet and in most instances this water never approaches the neutrality of pure distilled water.

As has been shown by the foregoing data, the use of distilled water in a laboratory test has demonstrated a great difference in the stability of various types of emulsions. It is entirely reasonable to assume that in the field when emulsions have been stored for some length of time, difficulties such as the formation of slugs in the spray nozzles may be encountered, or should alkaline waters be present, the asphalt may ball up when mixed mechanically. The tendency for some asphalt emulsions to coalesce and settle out has a marked relationship to the utility of that product in the construction field. The use of such material will mean that the coating of the aggregate will be non-uniform with the resulting loss in strength of the bond between the aggregate.

It has been standard practice for years to reject portland cement on account of objectionable lumps. Yet, there has been a tendency on the part of some chemists and engineers to pay little heed to the miscibility test on emulsions and in some instances to waive the settlement tests entirely, little realizing their significance in relation to field use.

The maximum difference in asphaltic content, as found in this modified test, does in each instance furnish some clue as to what may be expected as a settlement result. For instance, should the maximum difference be less than 4.1, it is safe to assume that the settlement test will meet the A.S.T.M. specifications.[1] Should the maximum difference be in excess of this value, the chances are that the 5-day results will not conform to specifications.

SUMMARY

It is evident from this preliminary work that there is some relationship between the behavior of diluted emulsions when tested by this suggested modified method and the probable 5-day results. The relationship is not fixed in any definite ratio which would permit the prediction of the 5-day results to an exact figure from the results of this modified test. The different type mills used by the various producers together with different types of bitumen, emulsifying agents, and the rate of production will all introduce factors that may prevent a direct ratio. However, on the basis of experimental data shown in this paper, it has been found that when the maximum difference in asphaltic content is less than 4.1, the 5-day settlement results have been in accordance with the present specified requirements. The converse is also evident that when the difference is higher than 4.1, the 5-day results have not been in accordance with the requirements.

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[1] Tentative Specifications for Emulsified Asphalt (for Coarse Aggregate Plant Mixes) (D 397 - 34 T), and for Emulsified Asphalt (for Retread and Coarse Aggregate Mixes) (D 398 - 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, pp. 916 and 918, respectively (1934); also 1934 Book of A.S.T.M. Tentative Standards, pp. 684 and 686, respectively.

Since it is highly desirable in the interest of both consumers and producers of emulsions to reduce the time necessary for laboratory release on material (this is evidenced by reduction this past year from a 10-day settlement test to a 5-day test), it is suggested that the method outlined be further studied by others. It is further proposed that this method be used as an alternate to the accepted 5-day settlement test and that the release of material be permitted should the results be less than 4.1 as mentioned above. In the event that the results were in excess of this amount, it would then be necessary to withhold release of material pending the outcome of the customary 5-day settlement test.

Acknowledgments:

In conclusion, the author wishes to express his grateful appreciation to the American Bitumuls Co., the Shell Petroleum Corp., the Bitucote Products Co., the Barber Asphalt Co., The Texas Company, and L. R. MacKenzie, Inc., for having cooperated and furnished samples that permitted this investigation.

In addition, the author is extremely grateful to E. E. Scholer, whose suggestions and comments were responsible for starting this work, and to F. W. Hoertel and Joseph L. Stewart whose aid and assistance in the laboratory made this work possible.

DISCUSSION

MR. J. S. MILLER, JR.[1] (*presented in written form*).—My first reaction to this paper is one of extreme interest because anything that can be done to shorten the time necessary to determine the properties of emulsified asphalts will certainly be appreciated by both consumer and producer.

I have never been in hearty sympathy with a settlement test, especially in connection with emulsions intended for mixing purposes, and in connection with the so-called quick-setting types I believe the most important feature is whether any segregation or migration of particles either to the top or bottom layer of the emulsion actually coalesce or remain as distinct dispersions. If there is coalescence of these particles in such a settlement test to the extent that they cannot be easily redispersed with mild agitation then of course such an emulsion cannot be used, but where such settled particles can be easily dispersed then I think the emulsion is a commercially usable one.

Mr. Downing uses the terms "stratify," "creaming," "stable," "coagulation" and "separation." He refers to "stratification" after dilution in the miscibility test which has been explained by some as an evidence of so-called "creaming," then relates "stability" to "stratification" and then questions what constitutes an "'appreciable' coagulation" or as it was called or stated in former years an "appreciable separation." This indicates the need for definitions of the terms used, a dilemma which faces all who are related to the emulsion field, and it is unfortunate that Mr. Downing did not attempt to undertake this task so that we could all be speaking in comparable terms.

I believe that stratification and creaming are related phenomena; also I believe that appreciable coagulation and appreciable separation are the same phenomenon but I do not believe that stratification and coagulation are necessarily related to each other unless during stratification the protective film of the particles is disrupted to the extent whereby particles coalesce and thus start coagulation. Stability of an emulsion can, of course, be related to coagulation but I do not believe stratification is related to stability.

I regret also that Mr. Downing has not given us those properties of the 69 samples examined which would give us an indication of which particular variety of emulsion was under examination. The footnote to Table I indicates he was considering the A.S.T.M. Specifications for Emulsified

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[1] Director, Technical Bureau, The Barber Asphalt Co., Maurer, N. J.

Asphalt (for Coarse Aggregate Plant Mixes) (D 397 - 34 T) and for Emulsified Asphalt (for Retread and Coarse Aggregate Types) (D 398 - 34 T) so in the absence of reference to emulsified asphalt (heavy premix—summer grade) (covered by A.S.T.M. Specifications D 399 - 34 T) we must assume that this very stable high asphalt content emulsion was not included. This is unfortunate in view of the fact that he speaks of the use of emulsions with wet aggregate and the D 399 - 34 T variety is peculiarly adapted to premix varieties where a high bitumen content must remain upon the aggregate and where normal aggregate dampness is to be taken care of. Consider for the moment what a *wet* aggregate means: A ton of aggregate containing 5 per cent of moisture will provide 12 gal. of water and assuming a mixture is to be made which should contain 2.5 per cent of bitumen and the emulsion contains 65 per cent of asphalt we will have 9 to $9\frac{1}{2}$ gal. of asphalt to the 12 gal. of water referred to above. A 5 per cent moisture content aggregate would present a difficult problem and is a greater amount of dilution than most commercial emulsions will stand without so modifying their viscosity as to have most of the emulsion flow from the aggregate.

I agree that emulsions can be adjusted with respect to viscosity to take care of small amounts of water in mineral aggregates, but I doubt whether any engineer can satisfactorily use a given type of emulsion with a mineral aggregate that is continually varying with respect to its water content.

Then, too, each aggregate when wetted presents a different problem, for some aggregates will not affect the usual type of emulsion stable to chemically active surfaces while other types of aggregates when wetted are extremely difficult to coat satisfactorily with the average type of emulsion. It has been the experience of most producers that a thorough knowledge of the anticipated aggregates in the field must be had to adjust production properly for such a condition.

The emulsion under A.S.T.M. Specifications D 399 - 34 T (heavy premix—summer grade) is peculiarly adapted to those situations where a high bitumen content is desired and yet there is no settlement test prescribed on this emulsion and it is unfortunate that Mr. Downing did not include this type of emulsion and definitely identify all of those he did include so that we could have had the benefit of this additional information.

My experience with the miscibility test dating back to the early days of the emulsion industry and even when it was first proposed has led me to believe it has a definite place in the testing of emulsion as indicative of an incipient coagulation or poorly dispersed asphalt originally, and this evidence as shown by an "appreciable separation" under this test taken in conjunction with the stone coating test has always been used to indicate the quality of the dispersion either from the standpoint of particle size or the efficiency of the protective film upon the particles.

If this proposed modification of the miscibility test is found to be all that Mr. Downing claims for it, I will be one of the first to advocate its very general inclusion in specifications.

MR. C. L. MCKESSON[1] (*presented in written form*).—Mr. Downing is indeed to be congratulated on his carefully prepared paper and for the work he has done in developing a new test method which may prove of great value. Mr. Downing makes it clear in his paper that his test method applies only to emulsified asphalt intended for mixing purposes and under no circumstances can it be applied to "quick setting" products, such as are described in A.S.T.M. Tentative Specifications D 401 – 34 T. In other words, he believes it is only applicable to a product which would be tested for "Miscibility with Water" under A.S.T.M. Tentative Methods of Test for Emulsified Asphalts (D 244 – 34 T).

The test method is presented as improved method of determining "miscibility" and also as an optional method determining probable "settlement" and these two uses will be discussed separately.

The present tentative test method is open to the objection that it is not quantitative. Rejection is made necessary if there is "any appreciable coagulation." Any test method which leaves final determination to the opinion of the operator should be made definite and quantitative if possible. The results of Mr. Downing's tests as shown in Table I indicate that coagulation which sometimes follows dilution and results in settlement within the 2-hr. period, can be quantitatively determined by comparing the percentage of residue obtained by top, middle and bottom strata. He does not suggest a maximum allowable difference, but Table I is significant in showing that no samples which passed the allowable limits in the settlement test failed in the present A.S.T.M. tentative miscibility test, while several samples passed present requirements for miscibility but failed badly in the settlement test. Coagulation results in increased particle size, which in turn accelerates settlement; it appears that the present visual method of determining coagulation is not entirely reliable. A maximum allowable difference for miscibility between strata of 4½, by the Downing method, would have eliminated all unsatisfactory samples which were shown as satisfactory by the present miscibility test. It is quite likely that different operators, working under the present miscibility test method, might have differed with the visual rating given some of the samples by Mr. Downing, but when measured quantitatively, such differences could not occur.

Mr. Downing's proposed test appears to have merit as a quick means for determining probable settlement and of preventing expensive and unnecessary delays in some instances. He apparently does not propose his test as a substitute for the present A.S.T.M. tentative settlement test method, but merely as an optional complementary test which could be used to justify immediate acceptance of some products. Failure by his method would merely necessitate waiting for the completion of the present tentative standard test before acceptance or rejection.

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[1] Director of Engineering and Research, American Bitumuls Co., San Francisco, Calif.

In the tests listed in Table I, acceptance of all samples having a maximum difference of 4 would have been justified by a settlement of less than 5, subsequently obtained by the tentative settlement test and in all of those instances would have eliminated delay.

Sometimes emulsified asphalt shows little tendency toward coalescence while new and fresh, but the stabilizer becomes ineffective after a lapse of time. Sample No. 61 in Table I might be an example of such a product. Although showing a difference of only 4.7 in the Downing test made in 2 hr., this product had a settlement of 14.45 (A.S.T.M. tentative test method) at the end of 5 days, probably produced by slow coalescence of the dispersed particles of asphalt.

The writer has had Mr. Downing's test method used in two laboratories under his direction by different operators, and the results checked satisfactorily with those obtained on the same samples by Mr. Downing.

Mr. Downing's test method deserves continued use in laboratories throughout the country as a parallel test to the present A.S.T.M. tentative test methods for miscibility and settlement with the hope that data may be obtained which will enable the committees responsible for these methods to embody its meritorious features in the present Tentative Methods.

MR. LESTER KIRSCHBRAUN[1] (*presented in written form*).—The author of this paper has directed his attention to a very pertinent annoyance in the methods of testing asphaltic emulsions for their ability to stay in suspension. Any method which would actually reflect the behavior of the material in service and make it possible to determine this within a reasonable time, would be a welcome substitute for the present 5-day sedimentation test.

It is assumed that the author is concerned primarily with the question of sedimentation of emulsions rather than with the usual miscibility test, which is employed of course in the determination of quite different properties. The miscibility test has for its purpose the determination of the ability of the material to withstand dilution without coagulation of the asphalt particles, regardless of what happens thereafter. The test is not applicable to the so-called quick breaking emulsions.

Where an emulsion actually will not withstand dilution according to the A.S.T.M. method there is usually very little difficulty in determining this visually. Where, on the other hand, a question exists as to the presence in the emulsion of coalesced particles or slugs, then the screen test which has been recently accepted as A.S.T.M. tentative, applies quantitatively and without difficulty.

The proposed test must be inapplicable to quick breaking emulsions because of the coagulating effect of diluting water, which would produce agglomerates or coalesced particles that would adversely affect the result of the test.

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[1] Vice-President, In Charge of Development, The Flintkote Corp., Rutherford, N. J.

In connection with the details of the method outlined by Mr. Downing, the position in the sample under test, from which the portions are withdrawn, is not given with sufficient exactness to be certain that results can be duplicated in different laboratories. It would be helpful if the depth of the various tubes in the liquid, either from the surface or from the bottom of the beaker, could be set forth.

The proposal to accelerate the rate of sedimentation by dilution so as to obtain results in one instead of five days, may be open to some question from a theoretical point of view. It might be argued that the condition of dilution does not generally represent the condition in which the material is received or used on the job. Then too, the rate of sedimentation is affected by the specific gravity of the two phases, and any change in this relationship necessarily introduces disturbing influences. Theoretically, two phases of asphalt emulsion having the same specific gravity should show no sedimentation over any period of time.

If, however, the specific gravity of the outer phase be reduced by dilution, an otherwise perfectly suspendable emulsion would tend to show a rate of sedimentation not inherent in the original material. Likewise, an emulsion in which the gravity of the asphalt phase is less than that of the aqueous phase, and one which would tend to cream on standing in its original condition, perhaps sufficiently to fail in the present 5-day test, might if diluted, tend to remain in suspension and possibly pass the proposed test. In fact certain tests made in our laboratories indicate this to be the case. The use of soap solution of a concentration such as to effect no change in the gravity of the outer phase might obviate this difficulty.

The results shown by the author indicate that an arbitrary figure can be set by his accelerated test which for the emulsions tested would, in a general way, pass materials that would pass the present A.S.T.M. 5-day test. Before such a figure could be established it would be well to have data concerning emulsions of the type which normally tend to cream.

It is unfortunate that neither the present 5-day A.S.T.M. test nor the proposed test has been related in a significant way to the actual behavior of the emulsions in service. Therefore the entire question as to whether the proposed test gives results similar to the one already in use is to a certain extent, an academic one. Certainly it will be very desirable to replace the 5-day test with one which will supply significant values within 24 hr. The test proposed should stimulate further effort in this direction.

MR. C. A. DOWNING[1] (*author's closure by letter*).—Mr. Miller has very properly raised a very pertinent point as to the confusion implied by the various terms mentioned in the introduction of my paper. All of these terms with the exception of "stratify" are ones that have been used by emulsion technologists. The meaning of the word "appreciable" when related to the miscibility test is something on which I can find no agreement among emulsion men and I heartily agree that some common basis of understanding should be reached on these terms. Since these terms have emanated mainly from representatives of the emulsion industry in the first place, it would hardly be proper for me to attempt to differentiate between them.

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[1] **Engineer in Charge, Municipal Testing Laboratory, St. Louis, Mo.**

I feel that some representatives of the emulsion industry have been prone in the past to confuse the phenomena of sedimentation and coagulation and have used these two terms as synonymous. On page 38 of "Modern Road Emulsions" published by The Carriers Publishing Co., Ltd., London, there is an excellent explanation of the two phenomena and the following excerpt will clearly explain the difference:

> "Sedimentation is in itself a reversible change and if it has not been followed by coagulation, there is no difficulty in re-distributing the phases of a sedimented emulsion by stirring or other suitable form of agitation. Sedimentation and coagulation are often used interchangeably, however, because, in most cases, where there is heavy sedimentation, coagulation sets in and appears to be a part of the sedimentation phenomenon.
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> "Sedimentation in an emulsion is the reversible separation of phases due to the action of the force of gravity on suspended particles. Coagulation is the irreversible separation of the two phases of an emulsion, due to coalescence to the bulk form, of the disperse phase. Intrinsically, the two phenomena are quite different and distinct, although, as has been said, heavy sedimentation is frequently followed by partial coagulation."

I believe that the above explanation will agree with Mr. Miller's statement regarding redispersion of the asphalt particles. However, I am of the firm opinion that should there be an excessive migration of asphaltic particles either to the top or bottom of the container the chances of coagulation are greatly increased with the resulting probable failure to meet the ordinary settlement requirements.

As stated in my paper, the work is based on diluting mixing emulsions with distilled water. I feel that this extreme dilution does serve to break down at least some of the protective layer around the asphalt particles and this accelerated test predicts in a fairly satisfactory manner the probable results which would be obtained should the asphalt be improperly dispersed, or should the emulsifying agent be of such a nature that it would change in character over a period of time.

Due to space limitations, I was unable to include the information regarding the characteristics of the 69 samples reported. There was quite a variety in the various types reported. Some of the manufacturers submitted their own particular grades of mixing emulsions and others submitted samples conforming to the types covered by A.S.T.M. Specifications D 397 – 34 T and D 398 – 34 T. The asphalt content of the samples tested ranged from 52 to 68 per cent. A large portion of the samples tested were emulsions of a far greater stability than the type mentioned by Mr. Miller. By stability, I mean inherent low 5-day settlement and in addition, ability to mix readily with fine material without breaking or balling-up. These latter type emulsions were intended to comply with the following requirements:

Viscosity, Saybolt Furol, 60 ml. at 25 C	not more than 100 sec.
Residue by evaporation (3 hr. at 163 C.)	55 to 60 per cent
Settlement, 5 days	not more than 5
Demulsibility, 50 ml. $0.1N$ $CaCl_2$	not more than 5.0 per cent
Sieve test	not more than 0.10 per cent
Miscibility with distilled water	no appreciable separation after 2 hr.
Portland cement mixing test	shall not show more than 5 per cent residue when 100 ml. emulsion is mixed with 50 g. of portland cement

The residue obtained by evaporation at 163 C. shall conform to the following requirements:

Penetration at 25 C	100 to 200, or 200 to 300
Soluble in carbon disulfide	not less than 97.5 per cent
Ash	not more than 2 per cent
Ductility at 25 C	not less than 60 cm.
Specific gravity at 25 C	not less than 1.00

The above type emulsion was one of several approved by the Board of Standardization in St. Louis almost eleven months prior to the publication of the present tentative specifications for emulsified asphalts. The extremely low demulsibility requirements together with the cement mixing test make this type of emulsion far more stable than the type mentioned by Mr. Miller and it is admirably suited for premixing stone that may contain a high percentage of fine material. After two years of very satisfactory use, we have considered it inadvisable to modify our emulsion specifications even though they are somewhat at variance with those recommended in the tentative specifications.

Lack of space also prevented the inclusion of additional data which indicated a growth in the particle size when the emulsions were diluted with distilled water. Micrographic evidence was obtained which clearly indicated that coagulation had taken place in the bottom of the beaker and there was a marked difference in size between the asphalt particles taken from the top and bottom of the beaker.

The points raised by Mr. Miller should react in a better understanding between producers and consumers. His sentiment towards the miscibility test is such that he should fully appreciate the modification proposed which will give a definite, quantitative value to a test which at the present time depends entirely upon the test operator's judgment. I am confident that after he has given my proposed method a thorough trial he will be convinced of its merit and the economic advantage that will naturally result to all those interested in this problem.

INVESTIGATIONS OF THE PHYSICAL PROPERTIES OF ASPHALTIC MIXTURES AT LOW TEMPERATURES

By L. F. Rader[1]

SYNOPSIS

This paper describes equipment and methods of test developed for determining modulus of rupture, modulus of elasticity in flexure, and toughness of asphaltic mixtures chilled to low temperatures. Modulus of rupture and modulus of elasticity in flexure are determined by testing a chilled beam of compressed asphaltic mixture on a 6-in. span. The toughness test is conducted by testing a chilled briquet in the standard Page impact machine. The specimens are chilled by using solid carbon dioxide as the refrigerant.

The test results show the influence upon physical properties of asphaltic mixtures at low temperatures of the following variables: (1) chilling temperature, (2) compaction of mixture, (3) percentage of asphalt, (4) asphalt of different origins, methods of manufacture, penetrations, and susceptibility characteristics. The stability at 140 F. of each mixture is given.

The behavior of asphaltic mixtures under load when chilled to a low temperature is discussed with respect to the cracking of asphalt pavements.

Among the conclusions it is pointed out that (*a*) sheet asphalt mixtures chilled to low temperatures are brittle materials having definite load-deflection curves, (*b*) the mixtures become stiffer and more brittle with decrease in temperature, (*c*) compaction is important to develop tensile strength at low temperatures, (*d*) the use of low normal penetration bitumen tends to make mixtures stiff and brittle at low temperatures, and (*e*) advantageous use of low-temperature physical tests of mixtures may be made in designing asphaltic mixtures.

INTRODUCTION

Considerable difficulty has been experienced with the cracking of asphalt pavements, particularly in the northern part of the United States and in Canada. It is true that many of the cracks occurring in asphaltic wearing surfaces are due to cracks in the concrete base, but some cracking is also due to the characteristics of the asphaltic mixtures themselves.

Although a great deal of attention has been given to the determination of the physical properties of asphaltic mixtures at normal and at elevated temperatures, particularly to the determination of stability, little work has been done on the physical properties of asphaltic mixtures at low temperatures. Quantitative data are available concerning the ability of asphalt paving mixtures to resist displacement, and methods of proportioning and selecting materials have been established, by means of stability tests, to overcome failure in service due to shoving, waving, and rutting. It is believed that these data need to be supplemented by the results of tests at low temperatures. Quantitative data obtained from low-temperature tests should aid engineers in solving problems relating to the control of cracking.

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[1] Assistant Professor of Civil Engineering, Polytechnic Institute of Brooklyn, Brooklyn, N. Y.

CRACKING OF ASPHALTIC MIXTURES

At normal temperatures asphaltic mixtures are plastic, that is, they deform under the continuous application of relatively small loads. When chilled to low temperature, the behavior of asphaltic mixtures under load is more nearly similar to that of such brittle materials as portland-cement concrete, stone, and cast iron. There is a definite load-deflection curve and significant increases in load are required to produce increased deformations. The change from plastic to rigid state appears to be a gradual one. At 0 F. ordinary sheet asphalt paving mixtures possess considerable rigidity and this rigidity is increased as the temperature is lowered.

Since this is the case, it seems reasonable to assume that the cracking of asphaltic pavements at low temperatures occurs in the same way as does the cracking of concrete pavements, namely, that it is due to excessive tensile stresses caused by contraction. Contraction is caused by reduction in temperature, and possibly by reduction in moisture content as has been indicated by some preliminary tests. In the case of mixtures of low voidage, temperature change is undoubtedly the more important factor. The tensile stress produced in chilled asphaltic mixtures due to temperature reduction may be calculated by the expression:

$$s = ctE$$

where s = tensile stress in pounds per square inch,
c = thermal coefficient of expansion per degree Fahrenheit,
t = total temperature change in degrees Fahrenheit, and
E = modulus of elasticity in pounds per square inch.

This stress is developed in the pavement if the edges and ends are restrained. If the edges are unrestrained, the length of pavement between cracks necessary to produce the frictional resistance between the pavement and its base needed to develop this stress may be calculated by the expression:

$$L = \frac{24\,s\,d}{f\,W}$$

where L = the length of the section of pavement between cracks in feet,
s = tensile stress in pounds per square inch,
d = the thickness of the pavement in inches,
f = coefficient of friction between the pavement and its base, and
W = weight of the pavement in pounds per square foot.

In planning the investigation of properties related to the above phenomena, the author has been primarily interested in obtaining values of the tensile stress and of the modulus of elasticity for asphaltic mixtures when chilled to low temperatures. Ideally, the tensile stress, *s*, and the modulus of elasticity, *E*, should be determined on a prismatic, or cylindrical, specimen by means of a tension test. From a practical standpoint, however, it is difficult to compact asphaltic mixtures so as to obtain uniform density from end to end or from top to bottom of the specimen. It is also difficult to grip the ends of a tension specimen composed of an asphaltic mixture.

FLEXURE TEST

The flexure test on specimens of asphaltic mixtures at low temperatures has many advantages over pure tension tests. Compaction of the mixture in forming beam specimens can be carried on in a manner approximating field conditions. Variations in density from top to bottom will also be similar to field conditions, if the load is applied on the top of the mixture in the forming mold. The effect of variations in density between the ends and the center of the specimen is minimized, since the beam fails in tension on the bottom side near the center of the span if a center load is applied. Modulus of elasticity in flexure as well as modulus of rupture can be obtained. In determining the modulus of elasticity, it is easier to measure deflections in the flexure test than elongation in tension tests. The values of the modulus of rupture and modulus of elasticity in flexure are not the same as tensile stress and modulus of elasticity in tension, but they are suitable physical properties for quantitative measurements and for comparison between mixtures of different ingredients and of varying proportions.

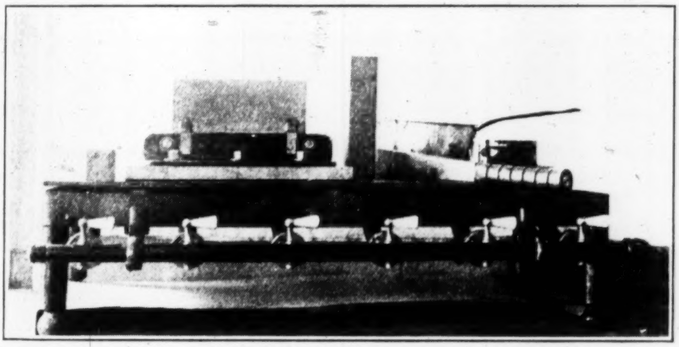

FIG. 1.—Equipment Used in Forming Beams of Asphaltic Mixture.

Molding the Beams:

The beams are molded in a steel mold, as shown by Fig. 1, consisting of a plate with angles forming the sides and ends bolted to it. Compression is applied to the specimen by means of a cold-rolled steel top plate. The compacted beams are 8 in. long, 2 in. wide, and 1½ in. deep. A depth of 1½ in. was selected as being typical of the thickness of sheet asphalt wearing courses. A length of 8 in. was selected so that a 6-in. span could be used in testing, thus giving a ratio of span length to depth of beam of four to one. The width was selected as 2 in. so that the tampers of the Hubbard-Field stability testing equipment could be used in compacting the mixtures.

Heating is done on a gas range. The mineral filler and sand are heated to a temperature of 350 F., weighed on a balance, and thoroughly mixed in a cylindrical mixing pan. The mixing pan is then placed on a balance and asphalt cement at 350 F. is weighed into the aggregate in the pan. The asphalt and aggregates are mixed for several minutes in the pan by means of a putty knife until the asphalt is thoroughly incorporated into the aggregate. An ironing action is then given the mixture by pressing it against the sides of the pan with the flat blade of the putty knife until all lumps are removed and a mixture of uniform characteristics is obtained. Approximately 900 g. of mixture are required for one beam.

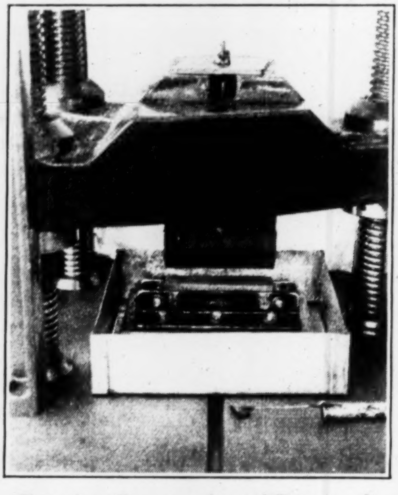

FIG. 2.—Compression of Beams of Asphaltic Mixture.

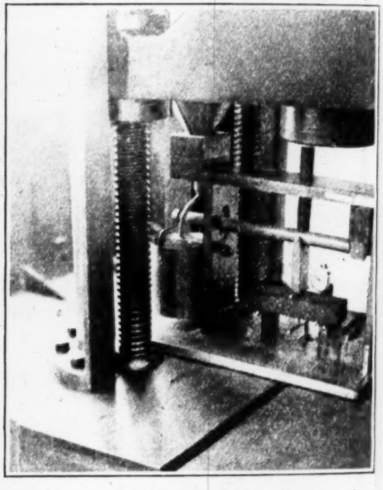

FIG. 3.—Equipment Used in Testing Sheet Asphalt Beams.

The mixture is placed in the steel beam mold through a galvanized-iron attachment mounted on the top of the mold to prevent loss of loose mixture. The mixture is then brought to a temperature of 250 F. and compacted by 100 blows from the blade tamper No. 1 and by 50 blows from the cylindrical tamper No. 2 of the Hubbard-Field stability testing equipment. Tamper No. 2 is fitted with a square base instead of a circular base. Care is taken to make the top surface level. The top plate is heated and placed on top of the compacted mixture. The mold is then carried by means of a handle to an empty galvanized-iron water bath set on the table of the testing machine.

The mixture is compressed at a temperature of 250 F. by applying load by means of the movable head of the testing machine through the top plate, as is illustrated in Fig. 2. A load of 3000 lb. per sq. in. is applied to the top surface of the beam, or 48,000 lb. total for the beam. The specimen is cooled for at least three minutes under load by pouring cold water into the water bath in which the mold is placed until the specimen is cooled to air temperature.

Testing the Beams:

The refrigerating box used for chilling the specimens is insulated with ground cork. Solid carbon dioxide, commonly called "dry ice," is used as the refrigerant. The author has been able to get a temperature of – 90 F. as measured by a spirit thermometer in this box. The dry ice is placed on a shelf in the top of the box and the specimens are placed on shelves underneath. The carbon dioxide gas settles to the bottom of the box, producing lower temperatures at the bottom than at the top of the box. Dry ice has been found effective in freezing specimens of asphaltic mixtures, but it is difficult to obtain definite temperatures, since the temperature varies with the amount of dry ice in the box. Specimens are kept in the box for at least two hours before testing.

The equipment for testing the beams is illustrated in Fig. 3. It consists of two roller supports set at a 6-in. span, a tool for applying load to the beam at the center, an arm for applying small loads, and a cylindrical bar for applying load from the testing machine to the specimen. The beam is placed on the roller supports with the original top side up. Loads up to 35 lb. are applied at the center by means of the small arm. Additional loads are applied at the center by the movable center tool of the testing machine at the rate of 0.05 in. per min. Deflections at the center of the beam are transmitted by means of a lever arm to an Ames gage of 0.0001 in. sensitivity. Deflection readings are taken corresponding to definite loads up to approximately 400 lb. load. Then the Ames gage and the lever are removed, and load is applied until the beam is broken. The breaking load is used for calculating the modulus of rupture. The load-deflection readings are plotted and the modulus of elasticity in flexure is determined.

The author has not been able to devise a satisfactory method for maintaining specimens at a constant low temperature during the time the specimen is being tested. The specimens increase in temperature when removed from the refrigerating box and placed in the normal air of the laboratory. In testing the specimens, the time of performing each operation, measured from the instant the specimen was removed from the refrigerating box, was recorded for each specimen. Temperature changes with respect to time after the specimen was removed from the refrigerating box were obtained by placing the thermometer in a hole in the center of a beam of asphaltic mixture. Temperature change curves were plotted by means of which approximate temperature corrections can be made.

Modulus of Elasticity in Flexure:

The modulus of elasticity for a beam tested in flexure under a load applied at the center is computed by the formula:

$$E = \frac{(P_2 - P_1)\, l^3}{48(\Delta f)I}$$

where E = modulus of elasticity in pounds per square inch,
P_2 and P_1 = loads applied at center in pounds,
l = length of span in inches,
Δf = net deflection at the center in inches, corresponding to load difference $(P_2 - P_1)$, and
I = moment of inertia of the cross-sectional area about the neutral axis.

The initial load, P_1, was taken at 35 lb. and P_2 at 335 lb. This method is equivalent to obtaining the modulus of elasticity by the secant method for a curved load-deflection graph, the secant being drawn between points on the curve corresponding to $P_2 = 335$ lb. and $P_1 = 35$ lb. The secant method is commonly used for determining the modulus of elasticity for materials such as portland-cement concrete and cast iron which have curved stress-strain relationships. The load-deflection graphs obtained for chilled sheet asphalt mixtures are curves, as may be seen by referring to Fig. 4.

Modulus of elasticity is a measure of the stiffness of the material. The greater the modulus of elasticity, the greater the stress set up in the material for a given change of length. From the standpoint of reducing cracking, it is desirable to have a low modulus of elasticity and a high modulus of rupture.

TOUGHNESS TEST

It is felt that an impact test to determine the toughness of asphaltic mixtures at low temperatures is needed in order to evaluate mixtures with respect to their ability to withstand the action of traffic. Toughness may be defined as resistance to impact. The author is using the standard Page impact machine, which is the equipment used for the test for toughness of rock[1] for determining toughness of asphaltic mixtures.

In performing the test, briquets of asphaltic mixture, 2 in. in diameter and 1 in. high, are molded in the Hubbard-Field stability testing molds. The directions given by Hubbard and Field[1] for forming briquets for the stability test, are followed. Sixty heavy blows with the No. 1 tamper (blade type) and 20 heavy blows with the No. 2 tamper (cylindrical type) are given the specimen at a temperature of 250 to 260 F. The briquet is compressed under a load of 3000 lb. per sq. in. and is cooled under pressure. The briquets are chilled in the refrigerating box using dry ice as the refrigerant for at least two hours as previously described for the beam specimens. A briquet, when chilled to the desired temperature, is placed with the original top end up in the Page impact machine on the anvil under the plunger. The height of the blow of the hammer in centimeters at failure of the briquet is taken as the toughness value.

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[1] Standard Method of Test for Toughness of Rock (D 3 - 18), 1933 Book of A.S.T.M. Standards, Part II, p. 1006.

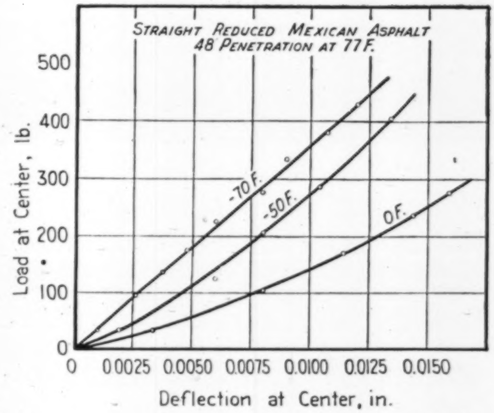

FIG. 4.—Load-Deflection Curves of Sheet Asphalt Beams at Various Low Temperatures.
Straight-reduced Mexican asphalt of 48 penetration at 77 F. Limestone dust filler. Crane sand.

DISCUSSION OF TEST RESULTS

The values of the modulus of rupture and modulus of elasticity of five beams of sheet asphalt mixture are given in Table I. This sheet asphalt mixture was a plant mix taken from uncompacted paving mixture laid on Kane St., between Court and Clinton Sts., in Brooklyn, on July 17, 1933. The approximate composition by weight was: asphalt cement, 10 per cent; limestone dust, 20 per cent; sand, 70 per cent. The asphalt was Mexican residual asphalt of 48 normal penetration at 77 F. The limestone dust was furnished by the New York Trap Rock Co., and the sand by the Crane Co. The stability of this mixture at 140 F. as determined using the Hubbard-Field stability testing machine was 2700 lb. It will be noticed that there is more variation in the values of the modulus of rupture than of the modulus of elasticity. Considering the variations due to temperature control in chilling the beams, it is thought that these values are fairly consistent.

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[1] Prévost Hubbard and F. C. Field, "The Rational Design of Asphalt Paving Mixtures," *Research Series No. 1*, The Asphalt Inst., November 1, 1934, pp. 6–13.

It should be noted that the specific gravity of all five beams was constant, indicating uniformity in molding the beams. The effect of variation in density on the value of the modulus of rupture is indicated in Table II. Increased density of mixture due to improved compaction greatly increases the value of the modulus of rupture, but apparently has little effect on the modulus of elasticity. It would appear from this limited number of tests that compaction of sheet asphalt mixtures is important to develop good tensile strength in the mixture at low temperatures.

TABLE I.—TESTS OF SHEET ASPHALT BEAMS.

Specimen	Temperature in Box, deg. Fahr.	Corrected Temperature When Broken, deg. Fahr.	Specific Gravity	Modulus of Rupture, lb. per sq. in.	Variation from Average, per cent	Modulus of Elasticity, lb. per sq. in.	Variation from Average, per cent
No. 1	−51	−19	2.19	1490	−3.5	234 000	+8.7
No. 2	−42	−10	2.19	1550	+0.4	216 000	+0.4
No. 3	−56	−30	2.19	1480	−4.1	212 000	−1.5
No. 4	−42	−16	2.19	1730	+12.0	207 000	−3.8
No. 5	−45	−16	2.19	1470	−4.8	207 000	−3.8
Average	−47	−18	2.19	1540	Avg. 5.0 Max. 12.0	215 000	Avg. 3.6 Max. 8.7

TABLE II.—TESTS OF BEAMS OF SHEET ASPHALT MIXTURE FROM KANE ST., BROOKLYN.

Specimen	Temperature in Box, deg. Fahr.	Specific Gravity	Modulus of Rupture, lb. per sq. in.	Modulus of Elasticity, lb. per sq. in.
No. 6	−58	2.17	1310	221 000
No. 7	−66	2.20	1990	205 000
No. 8	−3	2.17	900	153 000
No. 9	0	2.20	1620	153 000

TABLE III.—TOUGHNESS TEST OF SHEET ASPHALT MIXTURE FROM KANE ST., BROOKLYN.

Specimen	Temperature in Box, deg. Fahr.	Specific Gravity	Toughness
No. 10	−45	2.237	9
No. 11	−50	2.214	9
No. 12	−51	2.203	9
No. 13	−50	2.240	8
No. 14	−55	2.229	8

Values of the toughness of five briquets of the sheet asphalt mixture from Kane St., Brooklyn, are given in Table III.

Typical load-deflection curves are shown in Fig. 4. These curves illustrate the variation in stiffness of the mixture with respect to the temperature.

Variation of toughness of asphaltic mixtures with respect to temperature is shown in Table IV. This indicates that sheet asphalt mixtures tend to become more brittle as the temperature is reduced, and it may explain in a general way why certain mixtures deteriorate under the action of traffic in severe weather.

MIXTURES OF LOW ASPHALT CONTENT

The tendency of asphaltic mixtures of low asphalt content to crack has been pointed out by many observers and investigators. For instance, Mr. Prévost Hubbard[1] states:

"Frequently a high degree of stability in bituminous paving mixtures may be obtained by using a much lower percentage of bituminous cement than experience has demonstrated to be advisable. When this is done the mixture at low temperatures may possess most of the undesirable characteristics of a rigid structure."

The effects of reducing the percentage of asphalt in sheet asphalt mixtures upon stability, modulus of elasticity, modulus of rupture, and toughness are shown by Fig. 5. The characteristics of the mixtures have been accentuated by using a wide range of asphalt percentages. The results show that stability is increased by decreasing the percentage of asphalt, but that modulus of elasticity is increased and modulus of rupture and toughness decreased. It does not seem advisable to increase stability by reducing the percentage of asphalt below the amount required to produce compressed mixtures of low voidage, due to resulting increased stiffness and decreased tensile strength and toughness at low temperatures. Advantageous use of low-temperature physical tests as supplements to stability tests in designing asphaltic mixtures is indicated.

TABLE IV.—TOUGHNESS OF SHEET ASPHALT PAVING MIXTURE AT LOW TEMPERATURES.

Straight-reduced Mexican asphalt of 48 penetration at 77 F.

Asphalt Cement	Limestone Dust	Crane Sand	Voids	Stability at 140 F.
10.0%	16.5%	73.5%	2.6%	1960 lb.

TEMPERATURE, DEG. FAHR.	AVERAGE TOUGHNESS
+35	19.
−11	8.7
−50	7.5

EFFECTS OF VARIATIONS IN CHARACTERISTICS OF ASPHALTS

The effects upon the physical properties of sheet asphalt mixtures at low temperatures due to variations in characteristics of asphalt were investigated for six asphalts of 41 to 50 normal penetration produced from petroleums of different origins and by different methods of manufacture. The descriptions and physical properties of these asphalts are given in Table V. The stabilities and physical properties at low temperatures of these mixtures are shown in Fig. 6.

The results obtained indicate that for the Mexican straight-reduced asphalt, the steam reduced Mid-Continent flash-coil residue asphalt, the oxidized 70 to 80 F. softening point Mid-Continent flash-coil residue, the oxidized 71 F. softening point Colombian flash-coil residue and the oxidized

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[1] Prévost Hubbard, "Effect of Aggregates upon Stability of Bituminous Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 807 (1929).

115 F. softening point Colombian flash-coil residue asphalt there are no great differences in stability, modulus of elasticity and modulus of rupture.

The mixtures composed of oxidized Mid-Continent and Colombian asphalts show higher toughness values at low temperature than the mixtures composed of steam-reduced Mexican and Mid-Continent asphalts.

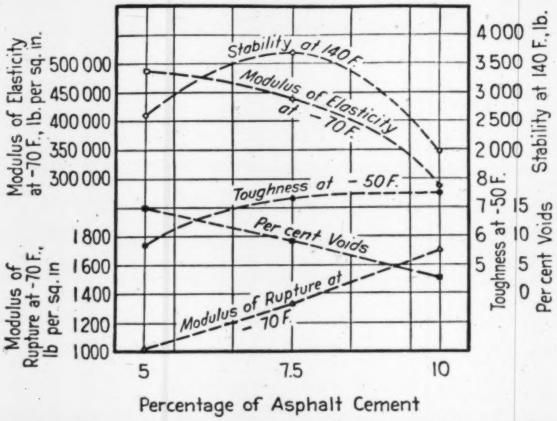

FIG. 5.—Effects of Variation in Percentage of Asphalt upon Physical Properties of Sheet Asphalt Paving Mixtures.

Straight-reduced Mexican asphalt of 48 penetration at 77 F. Constant ratio of limestone dust to sand for all mixtures.

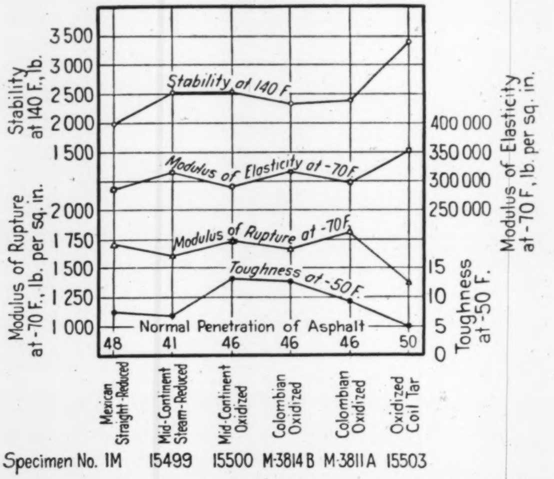

FIG. 6.—Effects of Variations in Characteristics of Asphalt upon Physical Properties of Sheet Asphalt Paving Mixtures.

Stabilities determined by means of the Hubbard-Field Asphalt Stability Testing Machine. Proportions of mixtures: asphalt, 10 per cent; limestone dust, 16.5 per cent; Crane sand, 73.5 per cent.

The oxidized cracking coil tar from gas oil feed is an asphalt according to standard definition[1] but is called cracking coil tar by many refiners.

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[1] Standard Definitions of Terms Relating to Materials for Roads and Pavements (D 8 – 33), 1933 Book of A.S.T.M. Standards, Part II, p. 1008.

It is very susceptible to temperature change. The mixture containing this asphalt shows high stability at 140 F. but high modulus of elasticity, low modulus of rupture, and low toughness at low temperatures.

EFFECTS OF VARIATION IN PENETRATION

Physical properties of sheet asphalt mixtures at low temperatures were determined with respect to variation in penetration of asphalt. Three types of asphalts were investigated: Mexican straight-reduced asphalt, oxidized 70 to 80 F. softening point Mid-Continent flash-coil residue, and oxidized cracking coil tar from gas oil feed. Cements of three different normal penetrations for each type of material were used. The physical properties of these asphalts are given in Table VI. The mixtures all contained 10 per cent asphalt, 16.5 per cent limestone dust, and 73.5 per cent Crane sand.

TABLE V.—PHYSICAL PROPERTIES AND DESCRIPTIONS OF ASPHALTS OF 41 TO 50 NORMAL PENETRATION.

Sample No. 1M produced by the Standard Oil Co. of N. J. Other samples produced by the Imperial Oil Refineries, Ltd., of Montreal.

	Sample No. 1M, Straight-Reduced Mexican Asphalt (Paving Grade)	Sample No. 15499, Steam-Reduced 95 to 100 F. Softening Point Mid-Continent Flash-Coil Residue	Sample No. 15500, Oxidized 70 to 80 F. Softening Point Mid-Continent Flash-Coil Residue	Sample No. M3814B, Oxidized 71 F. Softening Point Colombian Flash-Coil Residue	Sample No. M3811A, Oxidized 115 F. Softening Point Colombian Flash-Coil Residue	Sample No. 15503, Oxidized Cracking Coil Tar from Gas Oil Feed
Softening Point, deg. Fahr.	134	135	153	158	133	119
Penetration, 115 F., 50 g., 5 sec	190	130	87	93	180	too soft
Penetration, 77 F., 100 g., 5 sec	48	41	46	46	46	50
Penetration, 32 F., 200 g., 60 sec	13	12	26	27	28	9
Ductility at 77 F	110+	110+	6	6.5	110+	110+

TABLE VI.—PHYSICAL PROPERTIES AND DESCRIPTIONS OF THREE TYPES OF ASPHALTS OF DIFFERENT NORMAL PENETRATIONS.

Mexican asphalt produced by the Standard Oil Co. of N. J. Other samples produced by the Imperial Oil Refineries, Ltd., of Montreal.

	Straight-Reduced Mexican Asphalt			Oxidized 70 to 80 F. Softening Point Mid-Continent Flash-Coil Residue			Oxidized Cracking Coil Tar from Gas-Oil Feed		
	Sample No. 3M, 11 to 20 Normal Penetration at 77 F.	Sample No. 1M, 41 to 50 Normal Penetration at 77 F.	Sample No. 2M, 51 to 60 Normal Penetration at 77 F.	Sample No. 15502, 21 to 30 Normal Penetration at 77 F.	Sample No. 15501, 31 to 40 Normal Penetration at 77 F.	Sample No. 15500, 41 to 50 Normal Penetration at 77 F.	Sample No. 15505, 11 to 20 Normal Penetration at 77 F.	Sample No. 15504, 31 to 40 Normal Penetration at 77 F.	Sample No. 15503, 41 to 50 Normal Penetration at 77 F.
Softening Point, deg. Fahr.	165	134	127	222	171	153	136	123	119
Penetration, 115 F., 50 g., 5 sec	67	190	199	31	63	87	155	too soft	too soft
Penetration, 77 F., 100 g., 5 sec	16	48	68	22	35	46	17	40	50
Penetration, 32 F., 200 g., 60 sec	8	13	24	14	20	26	3	9	9
Ductility at 77 F	110+	110+	110+	1	2.5	6	110+	110+	110+

The results of these tests are shown by Fig. 7. Stability at 140 F. and modulus of elasticity at −70 F. increased with decrease in normal penetration for all three types of bituminous cements. This indicates that although stability against displacement is increased by lowering the normal penetration of the asphalt, the rigidity of these mixtures is increased at low temperatures.

The straight-reduced Mexican asphalt mixtures have good values of the modulus of rupture for all three normal penetrations investigated. The oxidized Mid-Continent asphalt mixtures have good values of the modulus of rupture for normal penetrations of 46 and 35 but a lower value for 22 normal penetration asphalt. The mixtures composed of the oxidized cracking coil tar from gas oil feed have relatively low values of the modulus of rupture for all three normal penetrations investigated.

The toughness of straight-reduced Mexican asphalt mixtures composed of 16 normal penetration asphalt is less than that of 48 and 68 normal penetration asphalts. The oxidized Mid-Continent asphalt mixtures all show relatively high values of toughness, but the values of toughness decrease with decrease in normal penetration. The oxidized cracking coil asphalt mixtures all have relatively low values of toughness.

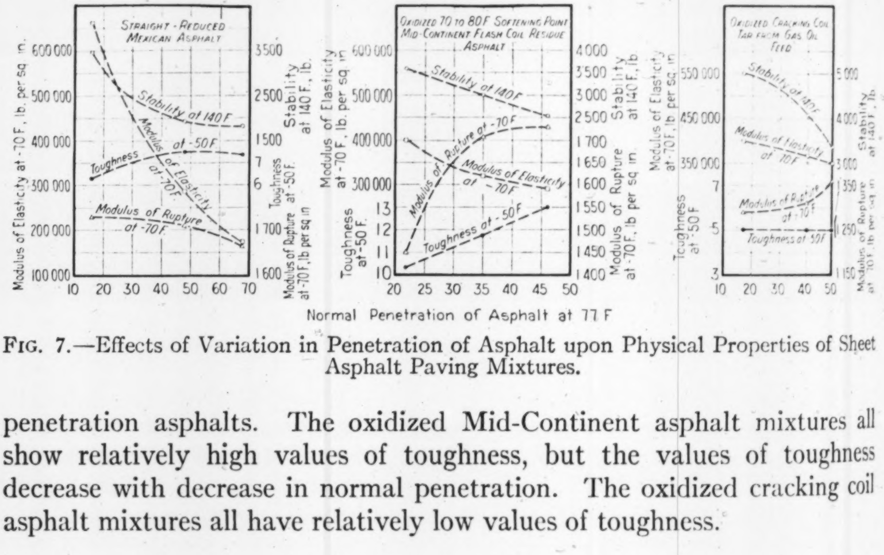

FIG. 7.—Effects of Variation in Penetration of Asphalt upon Physical Properties of Sheet Asphalt Paving Mixtures.

SUMMARY AND CONCLUSIONS

From the standpoint of reducing cracking in asphalt pavements, it is theoretically desirable for asphaltic mixtures to have low values of the modulus of elasticity and high values of the modulus of rupture. Modulus of elasticity is a measure of stiffness and modulus of rupture is a measure of tensile strength. It is also desirable for asphaltic mixtures to be tough, or resistant to impact.

Based on the results of physical tests made on sheet asphalt mixtures composed of Crane sand, limestone dust filler, and asphalts of various characteristics, the following conclusions may be stated:

1. Sheet asphalt mixtures chilled to low temperatures are brittle materials having definite load-deflection curves. The mixtures tend to become stiffer and more brittle with decrease in temperature.

2. Compaction of sheet asphalt paving mixtures is important to develop tensile strength at low temperatures.

3. Asphalt cement in sheet asphalt mixtures in amount required to fill approximately the voids in the mineral aggregate is desirable in order to decrease stiffness and develop tensile strength and toughness at low temperatures.

4. The mixtures composed of steam-reduced Mexican and Mid-Continent asphalts and oxidized Mid-Continent and Colombian asphalts of about the same normal penetration showed no great differences in stability, modulus of elasticity, and modulus of rupture, but these oxidized asphalts showed higher toughness values.

5. The mixtures composed of straight-reduced Mexican and oxidized Mid-Continent asphalts showed an increase in stability and modulus of elasticity and a decrease in toughness with respect to decrease in normal penetration. The mixtures containing oxidized Mid-Continent asphalt showed a decrease in modulus of rupture for asphalt of low normal penetration.

6. The mixtures composed of oxidized cracking coil tars from gas oil feed of three different normal penetrations showed high stability, but fairly high modulus of elasticity, low modulus of rupture and low toughness.

7. Advantageous use of low-temperature physical tests on asphaltic mixtures as supplements to stability tests in selecting materials and designing asphaltic mixtures is indicated.

Based on the results of tests illustrating the various characteristics of sheet asphalt mixtures, it would appear that the following indications have been developed relative to the effects of character, consistency and percentage of asphalt cement in these mixtures:

1. Asphalts of high susceptibility to temperature change, as illustrated by cracking coil asphalt, produce mixtures which are least resistant to cracking at low temperatures as indicated by their high modulus of elasticity, low modulus of rupture, and low toughness.

2. For the usual paving grades of asphalt, source and method of refining as covered by this investigation would appear to have little relation to resistance to cracking of sheet asphalt paving mixtures except that the oxidized asphalts in general develop somewhat higher toughness which should be advantageous from the standpoint of resistance to impact failure.

3. Other factors being equal, it would appear that those mixtures containing the highest penetration asphalt and the highest percentage of asphalt consistent with necessary stability should prove most resistant to cracking at low temperatures.

DISCUSSION

Mr. Prévost Hubbard.[1]—I wish to compliment Mr. Rader on the work that he has done in developing a method of evaluating asphalt mixtures in terms that the average highway engineer can understand and apply in his own work. So far, most of the work of and methods of investigators of bituminous materials have not conformed to the usual methods of evaluating mixtures to which the highway engineer has become accustomed. Mr. Rader's work should stimulate the further study of the mechanics of bituminous paving mixtures on the part of highway engineers.

His results seem to confirm the suspicion a good many of us have had as to the factors which influence resistance to cracking of paving mixtures. They appear to be quite logical and consistent.

Mr. Roland Vokac[2] (*presented in written form*).—Mr. Rader's paper is of unusual interest because it presents valuable new data in a field which has not been explored previously. Mr. Rader is particularly to be complimented on his engineering approach to the study of asphaltic mixtures on the basis of tests familiar to the engineer rather than solely on the basis of empirical tests, impossible of expression in engineering or scientific terms.

Mr. Rader has deliberately chosen to conduct his experiments at a temperature range somewhat lower than would be encountered under average field conditions in order that the elastic properties of the asphalt compounds might be accentuated at the expense of plasticity. This procedure has no doubt facilitated the determination of the elastic properties at the extreme low end of the service temperature range but has largely excluded the effect of temperature upon these properties. His data have great scientific value and are of practical interest in so far as they reveal the causes of cracking of pavements at low temperatures. It would, however, be unsafe to select asphalt cements or design paving mixtures solely upon the behavior of these materials at sub-zero temperatures.

Mr. Rader observed that high penetration and a generous percentage of bitumen are conducive to adequate elasticity at low temperatures. At the higher temperatures commonly encountered in service, both these conditions tend to increase the mobility of the pavement under the stress of traffic. The flow characteristics of the pavement at the higher temperatures tend to overshadow the elastic properties. It has seemed desirable to us to attempt a study of such properties as elasticity and toughness at temperatures which approximate the average temperate zone climate. The technic used in our adaptation of the Page impact testing machine to the testing of asphalt paving mixtures is described in a paper presented at the last meeting of the Association of Asphalt Paving Technologists.[1]

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[1] Chemical Engineer, The Asphalt Inst., New York City.

[2] Chief Engineer of Paving Research, Technical Bureau, Barber Asphalt Co., Maurer, N. J.

Briefly this test measures the energy of impact necessary to shatter a standard Hubbard-Field briquet in a single blow. Five or six briquets are subjected to blows from a given height, the height of blow being increased for each succeeding briquet. The height of impact and the number of blows to cause failure are recorded for each sample. These values are plotted on logarithmic paper and the height of blow which would cause failure in a single impact is determined by a simple extrapolation on a straight line through the points. This is shown in the accompanying Fig. 1. It will be seen that the slope of the straight line thus drawn is materially changed with the temperature of test. The slope is very slight at freezing temperatures and pronounced at ordinary temperature. This means that below freezing temperatures the mixture can sustain almost an infinite number of impacts provided they are of any degree of intensity less than that required to shatter the briquet in a single blow. On the other hand,

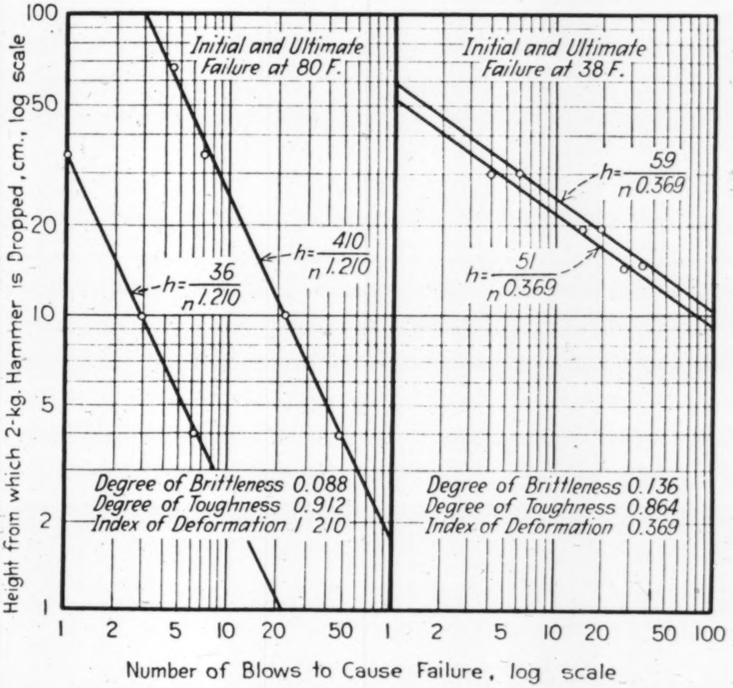

FIG. 1.—Relation of Height of Drop to Number of Blows to Cause Failure.

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[1] R. Vokac, "An Impact Test for Studying Characteristics of Asphalt Paving Mixtures," *Proceedings, Technical Sessions of the Association of Asphalt Paving Technologists*, January 30, 1935, pp. 40–48.

at normal temperatures the briquet will fail in a relatively small number of blows at impacts slightly lower than that required for failure in a single blow. In other words, the slope of the straight line indicates the degree to which a mixture can absorb impacts of less intensity than that required for failure in a single blow. This is of course a way to express the manner in which a mixture will resist failure. Theoretically, a slope of 0.00 indicates a material which will shatter like glass under a sufficient impact, while the opposite extreme, a slope of infinity, indicates a material that is like water which is so mobile and cohesive as to be impossible to shatter. Since asphaltic mixtures will tend to deform slightly under impacts less in intensity than required to shatter them in a single blow, the slope of this line is called the "Index of Deformation," *d*. The value of *d* for most of the mixtures we have tested thus far ranges from 0.0 to about 1.4. In the data from the same test we evaluate the impact resisting strength to impending failure and to total failure, the degree of brittleness and the degree of toughness.

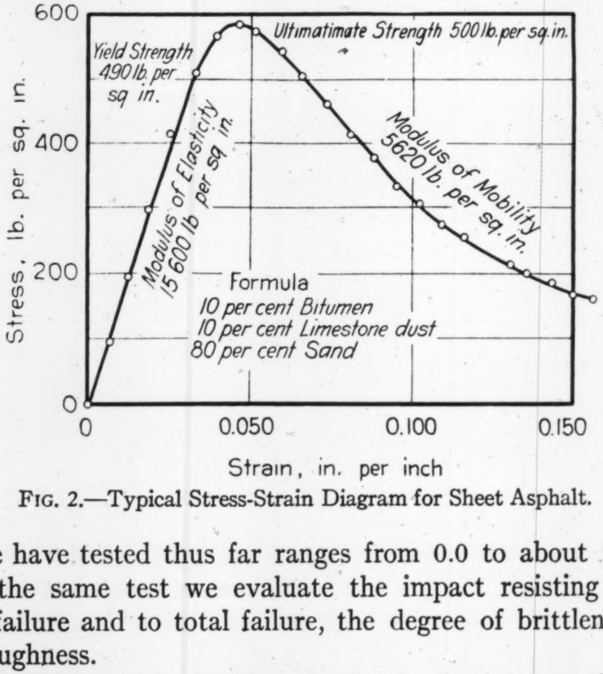

FIG. 2.—Typical Stress-Strain Diagram for Sheet Asphalt.

We have also found it possible to study the elastic properties of asphalt paving mixtures at the ordinary atmospheric range by the development of a proper technic for the use of the conventional compression testing machine such as would be used for example in the testing of portland-cement concrete. We find no difficulty in the development of stress-strain diagrams for compressed asphalt paving mixtures and have determined moduli of elasticity on a considerable variety of paving mixtures.

Our work to date has been of an exploratory character but we have already investigated to some extent (1) the effect of compaction, (2) the effect of variations in the composition of mixtures, and (3) the effect of asphalt cements from various sources.

The accompanying Fig. 2 is a typical stress-strain diagram produced in the compression test on a standard Hubbard-Field briquet such as we have used throughout all of our exploratory work. This test was conducted at room temperature—about 75 to 78 F. It can be seen that the first part of the stress-strain curve is a straight line, which definitely indicates that the asphalt mixture exhibits elasticity. This has been found invariably true in all of the tests. No visible lateral deformation appears in a specimen in the range of loads covered by this straight-line portion of the curve. The specimen bulges and assumes a barrel shape at higher loads, which becomes very pronounced as the load reaches the maximum point, and the whole sample finally tends to spread laterally as vertical deformation is continued. The first part of this last stage is also always represented by a straight line in the stress-strain diagram as shown in the line sloping downward to the right. The significance of this portion of the curve is still a little abstruse but is perhaps in a sense a measure of the mobility of the test specimen. For want of a better name we have tentatively called the slope of this line the modulus of mobility.

TABLE I.—EFFECT OF COMPACTION ON THE PHYSICAL CHARACTERISTICS OF MIXTURES.

Temperature of test, 78 F.

Compaction Load, lb.	Per cent Voids in the Mix		Modulus of Elasticity, lb. per sq. in.		Ultimate Strength, lb. per sq. in.	
	Mix A	Mix B	Mix A	Mix B	Mix A	Mix B
15 000	....	3.30	......	10 410	....	265
20 000	5.93	2.68	15 600	11 630	411	309
25 000	5.31	1.25	15 800	11 960	405	326
30 000	4.52	1.03	15 000	12 620	440	364
35 000	4.36	0.54	15 500	12 790	458	358
40 000	4.27	0.09	17 100	12 820	530	366
45 000	4.07	0.00	15 500	12 320	479	409
Average	....	....	15 750	12 080		

Briefly then, the factors measured by the compression test of asphalt paving are (1) the modulus of elasticity in compression, (2) the yield point, (3) the ultimate strength, and (4) the modulus of mobility. The first is measured in the straight-line portion of the stress-strain curve, the second is the load at which the stress-strain curve deviates from the straight line, the third is the maximum load recorded, and the fourth is the second straight-line portion of the stress-strain curve and has a negative slope.

Our studies with the compression test check Mr. Rader's findings that the degree of compaction has practically no effect on the modulus of elasticity. Briquets were compacted under various loads from 15,000 to 45,000 lb., the test results being shown in the accompanying Table I.

It will be seen that except for one discrepancy which is undoubtedly an experimental error, the modulus of elasticity does not vary appreciably.

Mix B, which is an unfilled mix with lower asphalt content shows some indication of lower modulus with the lighter compactions. However, when a fairly dense aggregate structure is attained as indicated in the lesser change in the voids with additional compaction, the modulus becomes again quite uniform. It will be noted, however, that the ultimate strength of the mixtures tends to increase with greater compaction in the same manner as Mr. Rader found the modulus of rupture to increase with compaction.

In mixtures holding the sand-filler ratio constant while varying the amount of asphalt cement, we find that Mr. Rader's conclusions are again substantiated in mixtures tested at room temperature. In the accompanying Table II are given the complete characteristics for a sheet asphalt mixture with several ratios of sand to filler.

TABLE II.—CHARACTERISTICS OF SHEET ASPHALT MIXTURES WITH VARYING SAND-FILLER RATIOS AND PERCENTAGE OF BITUMEN.

		8.0 per cent Bitumen	9.0 per cent Bitumen	10.0 per cent Bitumen	11.0 per cent Bitumen
Sand-filler ratio: R = 17	Percentage of voids in the mix	9.5	6.4	4.6	3.7
	Hubbard-Field stability at 140 F	1 640	1 560	1 270	1 010
	Modulus of elasticity in compression, lb. per sq. in	17 700	15 800	15 600	13 800
	Yield strength, lb. per sq. in	265	270	220	165
	Ultimate compressive strength, lb. per sq. in	345	370	320	275
	Modulus of mobility, lb. per sq. in	3 550	3 250	2 500	1 900
Sand-filler ratio: R = 8	Percentage of voids in the mix	7.9	5.3	3.9	3.2
	Hubbard-Field stability at 140 F	1 750	1 610	1 100	890
	Modulus of elasticity in compression, lb. per sq. in	21 400	18 600	14 100	12 950
	Yield strength, lb. per sq. in	350	290	190	135
	Ultimate compressive strength, lb. per sq. in	420	370	290	240
	Modulus of mobility, lb. per sq. in	4 550	4 000	2 350	1 600
Sand-filler ratio: R = 5	Percentage of voids in the mix	7.3	5.1	3.9	3.0
	Hubbard-Field stability at 140 F	1 860	1 700	990	700
	Modulus of elasticity in compression, lb. per sq. in	21 600	18 800	14 900	14 000
	Yield strength, lb. per sq. in	395	340	220	175
	Ultimate compressive strength, lb. per sq. in	520	425	295	250
	Modulus of mobility, lb. per sq. in	4 800	4 200	2 550	1 850

Many interesting conclusions may be drawn from these data.

Our investigation of the effect of asphalts from various sources has been of such a fragmentary character we will not attempt to present our data in tabular form. Suffice it to state that we have found significant differences in the various properties developed during the compression tests when testing mixtures in which the type of asphalt is the only variable.

Our experiments in the adaptation of the compression machine to the testing of asphalt paving mixtures have been performed through the courtesy of the manufacturer of the Baldwin-Southwark hydraulic compression machine developed for the testing of portland-cement cubes.

MR. C. M. BASKIN[1] (*by letter*).—Prior to 1922, the all-important question was how to prevent sheet asphalt pavements from shoving or deforming under traffic loads at high summer temperatures. This problem was ably handled by Mr. Prévost Hubbard with the result that, generally, little if any difficulty is encountered in this direction. On the other hand, we are now faced with difficulty at the other extreme, cracking of sheet asphalt pavements at low temperatures.

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[1] **Asphalt Technologist, Imperial Oil, Ltd., Toronto, Ont., Canada.**

The work carried out on behavior of dense asphaltic mixtures at high temperatures will need to be paralleled by a study of the behavior of the same mixtures at low temperatures. As such, Mr. Rader is to be highly commended, first, for his approach to the subject, and secondly, for his courage in undertaking a problem which many have frequently spoken of, but none dared undertake. This reluctance to undertake an investigation of cracking of sheet asphalt pavements was, no doubt, due to the fear that by correcting the situation at one end, it may have aggravated it at the other. Mr. Rader's work definitely shows that the more rigid a material, the more it will crack. While past investigations on characteristics of sheet asphalt mixtures at high temperatures mainly concentrated on arriving at factors whereby these mixtures can be made to have low mobility or approach a solid state at elevated service temperatures,—factors such as grading of aggregate, filler, penetration limits, percentage of bitumen, etc.,—apparently what impressed the paver most was low penetration; if a 60 penetration asphalt cement gave trouble with shoving, it was reduced to 50 or 40. Reducing content of asphalt was also a favorite means of raising stability.

As the basic cause for cracking of sheet asphalt mixtures is low temperatures, the farther north we go, the more severe is cracking, with this exception, that in most northern areas, low winter temperatures are not necessarily accompanied by similar mitigation of summer temperatures. We in Canada, therefore, have a special interest in this subject, and have consequently made considerable laboratory and field observations. Any extensive laboratory investigations should be preceded by careful field observations, in relation to the technical history of the materials used in construction of these pavements.

First of all, what are the critical temperatures as far as cracking is concerned, or at what time of the year is the pavement more likely to crack? In this connection it was repeatedly noticed that cracking does not necessarily occur during the time of the year when the pavement is at its lowest temperature. Rather is cracking most noticeable during the spring when pavement temperatures range all the way from 25 F. to 45 F. Time and again we find pavements subjected to temperatures of −30 F. or −40 F.—and clear of snow—showing hardly any cracks. The same pavements examined when frost starts coming out of the ground show the appearance of cracks in ever-increasing number. Portions of the same pavement covered with snow are comparatively free from cracks. If our observations are correct, it may well indicate the possibility that contraction plays very little part in the formation of cracks; otherwise most, if not all, the cracks would occur when the pavement is at its lowest temperature.

Also, it is questionable whether sudden traffic impact when the pavement is in a rigid condition is responsible for cracking. If it were, then cracking would again be more prevalent during the winter than during the spring. The fact that most of the damage occurs during the period when frost is coming out of the ground would lead one to suspect that the slow and gradual strains caused by the movement of subsoil may well be the most potent cause of cracking. All this would reduce our problem largely to pliability of paving mixtures at 32 F.

Our field observations further show definite signs that whenever there was lack of control of mixing temperatures, subsequent cracking was decidedly on the increase. Moreover, cracking of such pavements gets more severe with time. Plant temperature control emphasizing low mixing temperatures invariably results in a marked decrease in cracking. On the other hand, wherever mixing temperatures were unduly high, there was severe cracking, not only within the first year the pavement was in service, but becoming more aggravating from year to year. Also high plant mixing temperatures with asphalt spread out in fine films is an ideal condition for hardening by oxidation. Asphalt, for instance, can be raised materially in consistency by exposure of a fine film to the atmosphere, maintaining a high reaction temperature. The lower the mixing temperatures, therefore, the less chance there is of hardening of asphalt cement by oxidation.

That oxidation of asphalt frequently takes place during the process of mixing with superheated mineral aggregates is evidenced by the fact that extracted asphalt cement from overheated paving mixtures shows softening point - penetration relations characteristic of oxidation, rather than hardening due to the application of direct heat.

It is further interesting to note that in tracing the history of cracked and uncracked pavements, we are particularly impressed with the fact that wherever there is a reasonable amount of traffic and especially if the pavement was constructed during warm weather making possible better spreading and compaction, the asphalt cement may have hardened considerably by high mixing temperatures, yet gives little trouble with cracking.

My reason for entering into the above discussions and particularly in elaborating on field and laboratory observation is that the results of these observations are very pertinent to Mr. Rader's investigation. For instance, should we be able to establish that the critical temperatures, as far as cracking is concerned, are 32 F. to 45 F., a good deal of the laboratory difficulties with work at extremely low temperatures is eliminated. If we can establish that contraction has little if any effect on cracking, it becomes entirely a matter of toughness and pliability at critical temperatures.

If we are correct in regard to effect of high mixing temperatures on hardening of asphalt cement, and especially that even a 32 penetration asphalt cement when well compacted gives no trouble with cracking, it further simplified our problem. This would definitely indicate that control of mixing temperatures to a point where we know that, starting with a 56 penetration asphalt, the asphalt in the mixture when spread out and compacted is approximately of the same consistency, trouble with cracking will be materially reduced, if not eliminated.

We propose to continue our field and laboratory investigations in an attempt to classify and arrive at factors responsible for cracking. So far, our observations definitely substantiate Mr. Rader's findings and conclusions. Greater toughness of mixture reduces, and in many cases eliminates, cracking as evidenced by the relative absence of cracks on a heavy traffic pavement, compared to considerable cracking on light traffic pavements made with identically the same mixture.

For any given penetration asphalt cement, the lower the susceptibility of the asphalt cement to temperature change, the less cracking.

I therefore feel that, to supplement work such as carried out by Mr. Rader, technologists interested in the problem should do everything possible to accumulate facts and data on sheet asphalt pavements, both in the field and laboratory, from which a classified technical history could be reconstructed. Such data would include the following:

1. Degree of cracking *versus* time of pavement under service.
2. Hardening of asphalt cement *versus* temperature and mixing period.
3. Character of bitumen and nature of aggregate used.
4. Degree of cracking *versus* period of year, which would require information wherever possible as to when appearance of cracks was most noticeable and approximately what the temperatures were.
5. Degree of cracking *versus* penetration of asphalt at 77 F.
6. Cracking *versus* subsoil movement; taking a street or boulevard on which the central portion is kept completely free of snow, is there any marked freedom of cracks on the portions covered by snow as against the central portion free of snow?

The information, or a good deal of it, is there. It is merely a matter of collecting and compiling available data.

MR. L. F. RADER[1] (*author's closure by letter*).—The author appreciates very much the additional information regarding the effect of compaction and the effect of variations in the composition of mixtures contributed by the discussion of Mr. Vokac. The results of these investigations at room temperature substantiate the results obtained by the author at low temperatures. This would appear to indicate that mixtures designed to resist cracking at low temperatures would have characteristics tending to resist cracking at higher temperatures.

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[1] Assistant Professor of Civil Engineering, Polytechnic Institute of Brooklyn, Brooklyn, N. Y.

The discussion of the cracking of asphalt pavements by Mr. Baskin is a valuable contribution to the subject. Mr. Baskin states that 32 F. may be the critical temperature for the determination of pliability since strains caused by the movement of subsoil when frost is coming out of the ground may well be the most potent cause of cracking of asphalt pavements. If a mixture is designed to have a relatively low modulus of elasticity at low temperature, it would seem that it would have a relatively low modulus of elasticity, or a high degree of pliability, at 32 F.

The temperatures used in the investigation were low enough to produce an elastic state in the asphaltic mixtures composed of paving cements of ordinary consistency. Asphaltic mixtures in the elastic state can be tested by methods such as are used for brittle materials. Test values were obtained near the extreme lower end of the temperature range apt to be encountered in the field and supplement stability test values determined at 140 F. which is near the upper limit of temperature.

The investigation dealt with the characteristics of asphalt paving mixtures as they existed at the time of testing, the effects of variations in plant and street construction operations not being considered. The field observations given by Mr. Baskin in regard to these operations indicate the importance of proper inspection and control.

Mr. Baskin refers to the effect of traffic in reducing cracking of asphalt pavements by increasing the toughness of the paving mixture. In this connection, the following statement by Mr. Clifford Richardson[1] is pertinent:

> "Traffic releases tension to a large degree in a cold asphalt surface and assists elongation of the bitumen, so that heavy-traffic streets do not crack as readily as those of light or no traffic, and oftener crack only in the gutters, if at all, while light-traffic streets crack entirely across the roadway."

The accumulation of data such as Mr. Baskin suggests and correlation of it with results of laboratory tests should be carried on to provide more information concerning the resistance of asphalt paving mixtures to cracking.

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[1] Clifford Richardson, "The Modern Asphalt Pavement," Second Edition, p. 484.

A TEST PROCEDURE FOR PLASTIC CAULKING MATERIALS[1]

By D. W. Kessler[2]

Synopsis

Plastic caulking materials comprise a class of products which has, during recent years, aided materially in overcoming one of the shortcomings of masonry buildings. Although the sealing of joints between masonry and door or window frames constitutes a large part of the use of the plastics, there are numerous other similar purposes which they serve in building construction.

Being a comparatively recent addition to the list of building materials, there is much to be learned about the composition, fabrication and control of these products for the varied service conditions to which they are exposed.

A few years ago the National Bureau of Standards was confronted with the task of devising a means for determining the quality of these materials. The nature and uses of the product were such that tests or equipment commonly used on other types of building materials were not applicable. New test procedures had to be developed and the necessary testing apparatus designed.

Four types of tests are now used and are intended to determine qualities having a bearing on service performance. They have been in use for four years and a large number of samples have been tested for use on government jobs. The "strain test" determines whether the material remains sufficiently plastic in a joint to accommodate itself to structural movements which vary the width of the joint; "staining tests" determine whether the plastics discolor masonry near the joint and also give information on the performance of the material; "slump tests" determine whether the materials will become too soft in summer and flow out of the joints; and "copper corrosion tests" indicate certain undesirable properties of the plasticizing ingredients.

Introduction

The materials referred to in this paper are intended mainly as fillers for joints which are apt to vary in width, owing to settlement of the structure, thermal expansion and contraction, or other causes. They are of a plastic nature and are supposed to remain plastic over a considerable period of time. Although a few manufacturers have been making such materials for 25 years or more it is only during the past 10 years that the products have come into extensive use. During this period numerous trade brands have been placed on the market.

In masonry structures these plastics serve a useful purpose as fillers for expansion joints and for sealing the spaces between window or door frames and the adjoining masonry. In steel or concrete frame construction they are of value in sealing the joints between the spandrel beams and walls. Occasionally they have been used on stone masonry for general pointing purposes, and they are frequently employed in the vertical joints of copings, cornices, etc. They might serve a useful purpose also in wood structures, particularly to prevent the entrance of moisture at joints inaccessible for painting.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Chief, Stone Section, National Bureau of Standards, Washington, D.C.

COMPOSITION

The products of different manufacturers vary considerably in composition. In general they consist mainly of inert fillers or pigments reduced to a plastic condition with non-drying or treated drying oils. A variety of fillers are used such as whiting, asbestos, zinc oxide, zinc sulfide, barium sulfate, white lead, titanium oxide, silica, calcium sulfate, iron oxides, carbon, etc. A common mixture consists of whiting and asbestos mixed with a suitable oil and a small amount of volatile thinner such as mineral spirits. Some preparations include asphalt, rosin or rubber in the mix. There seems to be a trend among manufacturers toward the use of fillers more stable than whiting, but practically all formulas contain asbestos. Several products contain a mixture of various filling ingredients. Examination of chemical analyses of various products over a period of years will show wide variations in composition, which may indicate insufficient care in factory control.

PHYSICAL PROPERTIES

Certain physical properties are necessary besides that of plasticity. The caulking must adhere to the materials; it must have proper working qualities; and above all it must have the power of retaining its oil in order to remain plastic when placed in contact with the masonry materials, most of which tend to draw the oil from the compound by capillary forces. If an appreciable amount of oil is so removed the adjoining masonry is discolored and usually the caulking material shrinks, cracks, and becomes hard. Although some manufacturers have had considerable success in overcoming this difficulty, it appears that factory control has not been such as always to insure freedom from this so-called "bleeding."

Caulking compounds are usually marketed in such a plastic state that they can be forced through a metal tube of small diameter and are called the "gun grade." Materials of stiffer consistency that have to be applied with a hand tool are called the "knife grade." The gun grade can be applied more easily and economically and it usually has better adhesive qualities than the knife grade. However, unless the nozzle of the gun is small enough to enter the joint the caulking is not apt to fill the opening as satisfactorily as when applied with a hand tool.

The durability of the material should be considered as that period during which it retains sufficient plasticity and adhesiveness to maintain a tight joint under structural movements. In tests of joints between limestone slabs there have been a considerable number of failures at the end of 30 days with no movement of the slabs. On the other hand, some manufacturers refer to installations which are said to be in good condition after 15 or more years of service.

Caulking compounds apparently do not suffer greatly from the usual weathering agencies which cause deterioration of mortars, glazing putties, or paint. Ordinarily they are dense and water repellent. If oxidizable oils are used they must be pretreated to overcome oxidation in the finished product. The most common cause of failure in service is probably that a part of the oil is absorbed by the materials with which it is placed in contact.

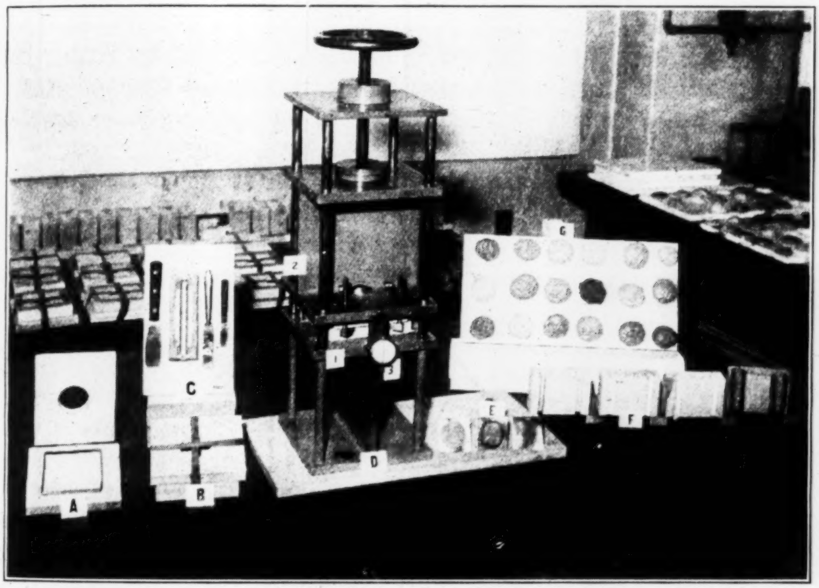

FIG. 1.—Apparatus and Test Specimens Used in Testing Plastic Caulking Materials.

TESTING OF CAULKING MATERIALS

The testing procedure developed and now being used at the National Bureau of Standards is intended to show the behavior of the materials under conditions simulating as nearly as feasible those of actual installations. The tests involve placing the caulking in contact with limestone, marble, and copper. Grade *B* Indiana limestone and any white marble are used. Both types of stone are thoroughly dried before preparing the test specimens, so as to obtain uniformity. This drying probably increases the capillary action on the oils. The various tests are described in detail as follows:

Strain Test:

The purpose of the strain test is to determine whether the caulking remains sufficiently extensible and adhesive to maintain a water-tight joint when subjected to strain.

Specimen.—The joint to be tested is made between two slabs of limestone 6 by 7 by 1 in., one of which has a hole in the center about 2½ in. in diameter (Fig. 1, *A*). The slabs are spaced ¼ in. apart by a strip of brass ¼ by ⅛ in. in cross-section, bent to form a square 4½ in. on the side. This spacer is laid concentrically on one slab and the other slab is then placed on top so that the long side of one slab is perpendicular to the short side of the other. This forms ½-in. projections by means of which the specimen can be clamped in the straining apparatus (Fig. 1, *B*). The slabs to be caulked are clamped on the spacer and the caulking is forced in at the four edges, giving a joint ¼ in. thick and ¾ in. deep. Two strong rubber bands are placed around the specimen and it is cured at room temperature for thirty days. The tools used in preparing the various specimens are shown at *C* in Fig. 1.

Apparatus and Test Procedure.—The apparatus shown at *D* in Fig. 1 was designed for this test. It consists of a base section, 1, rigidly fastened to four vertical rods which serve as guides for the movable section, 2, which can be raised or lowered by means of the screw shown at the top.

Before placing the specimen in the apparatus the space between the slabs and the caulking is filled with water and more water may be added, during the test, through the hole in the center of the upper contact plate. Figure 1, *D*, shows the specimen in position for testing; the lower stone slab is clamped to section 1 and the top slab to section 2. The slabs are separated by increments of 0.01 in. at approximately 15-sec. intervals and the joint is watched for leaks. A gage shown at 3, reading to 0.001 in., measures the amount of separation of the slabs as section 2 is raised. The present requirement is that the joint shall stand a strain of at least 0.05 in. before failure.

Slump Test:

The slump test is intended to determine whether the material is too soft to remain in a vertical joint.

Specimen.—The specimens shown at *F* in Fig. 1 are blocks of limestone approximately 4 by 4 by 2 in. with two parallel grooves ⅜ in. wide and ¾ in. deep. The surfaces of one groove are coated with lacquer or a solution of aluminum stearate to seal the pores. This treatment is applied at least 24 hr. before the grooves are caulked. Since this specimen is also used for the staining test it is important that none of the caulking touches the face of the stone. This is prevented by the shield shown at *C* in Fig. 1.

Test Procedure.—The specimen is supported as illustrated so that the caulking compound is free to flow out of the joint. It is left in this position for 24 hr. at room temperature and then transferred to an oven where it is exposed to a temperature of 50 C. for 24 hr. The requirement is that there shall be no appreciable slumping of the material under these conditions.

Staining Tests:

The purpose of the staining test is to determine whether oil will be drawn from the compound when placed in contact with limestone or marble.

Test Procedure on Limestone.—The specimen used for the slump test also serves for the staining test, it being inspected at the end of the 30-day curing period. The penetration of oil into the stone is readily seen by the discoloration it causes. One specimen (Fig. 1, *F*) illustrates a pronounced case of "bleeding," while another specimen shows no staining.

Test Procedure on Marble.—For this test a pat of the material is placed on a slab of white marble, and observations are made after 30 days to determine whether oil spreads around the pat. Several specimens are shown in Fig. 1, at *G*, some of which show the spread of oil through the pores of marble.

Copper Corrosion Test:

A test for the quality of the oil in the caulking material is made in the copper corrosion test, the presence of free fatty acids or sulfides being indicated by discoloration of copper.

Specimen and Test Procedure.—The specimen is made by placing a small pat of the sample on a bright copper plate. This is allowed to stand in the laboratory for 5 days, when the pat is scraped off and the plate cleaned with benzol. Corrosion of the copper is indicated by discolorations which are usually brown or black. The plates may be used numerous times by scouring the surface with a fine grade of steel wool before making the specimens. A specimen is shown at *E* in Fig. 1, also a plate which was discolored in the test, and another which was not.

DISCUSSION OF TEST RESULTS

The strain test, after 30 days curing of the specimen, gives information only for the initial stages of curing. In connection with an investigation now in progress, specimens are placed out-of-doors for a year before applying this test. However, a considerable percentage of the samples now being submitted for Government work fail in the strain test (within 30 days) and when this test was first put into use about two-thirds of the samples failed; many leaked as soon as water was poured into the specimens. Failures of this kind are due to shrinkage of the caulking and poor bonding properties. Usually the shrinkage is caused by removal of oil from the sample by the stone. The materials may be expected to give better results in contact with less absorbent masonry. The oven temperature (50 C.) used in the slump test is assumed to be the maximum attained on masonry walls in summer. Some formulas contain ingredients which greatly increase the tendency of the materials to flow at higher temperatures and the test is of value in showing such effects. Treatments applied to seal the pores of the stone usually increase the tendency to slump.

Staining tests were designed to determine whether the caulking material would produce undesirable effects on the masonry, but they were also found to be of considerable value in showing how well the caulking will serve its purpose. Where equipment is not available for complete tests, the staining test will serve to give valuable information and next to the strain test is believed to be the most determinative of quality. It can be made on any masonry material as described above in the test on marble, hence it can be used on the job. Dense materials like granite and marble are more apt to stain by drawing oil from the caulking material than are more porous masonry materials. However, a slight spread of oil from the caulking compound into the dense materials may not cause appreciable injury to the compound because it requires a very small amount of oil to fill the pores of such materials. The application of a lacquer or aluminum stearate solution frequently overcomes the tendency of caulking materials to stain the masonry and probably increases the life of the caulking.

The copper corrosion test is required by some specifications but little is known of its value. It is apparently based on the belief that sulfonated oils or oils containing fatty acids are objectionable. A large percentage of the samples pass the test satisfactorily. Usually samples that corrode copper also fail in some of the other tests, but samples that do not corrode copper may likewise fail in other tests.

The $\frac{1}{4}$-in. joint employed in the strain test was arbitrarily chosen as being representative of the joints in cornices and copings. Some joints in buildings may be $\frac{1}{2}$ in. or more in width. No studies have been made to determine whether a thick joint is more or less favorable to the caulking material. The $\frac{3}{4}$ in. depth of joint in the specimen used in the strain test is probably greater than that obtained on buildings with gun application, and in this respect the test may be too favorable to the caulking material.

It may be assumed that usually some moisture is present in masonry walls when caulked, and this may be favorable to the caulking by overcoming to some extent the capillary action on the oils. However, considerable moisture might affect adhesion adversely. As the specimens are always dried before caulking, for uniformity, the test probably represents more severe capillary conditions than usually occur in actual service, but bonding in the test might be expected to be more favorable.

The problem of securing a uniform masonry material for the specimens is difficult. Limestone was chosen because it is representative of a large part of the building stone in service. The grade employed is probably near the average of masonry materials in porosity and capillary effects. There is an appreciable variation in this grade of limestone and a more uniform material would be desirable. The marble used in staining tests should probably be standardized although there does not seem to be much variation in marbles from different sources.

Reference has already been made to a study on caulking materials now in progress at the National Bureau of Standards. The program includes the study of the behavior of representative samples on limestone, marble, brick, wood, and steel. Specimens are to be exposed to the weather for at least a year before testing, and particular attention is being given to the loss of oil from the caulking in contact with various materials. It is hoped that this investigation will afford information of value on the performance of caulking materials and form a basis for better methods of testing.

DISCUSSION

Mr. R. D. Moore[1] (*presented in written form*).—It is gratifying to know that some work has been done toward devising suitable methods of tests on caulking compounds. The tests described in the paper by Mr. Kessler are interesting and appear to be a proper approach to the subject. But the paper would have been of still more interest if some mention had been made of tests of a more accelerated nature.

The paper indicates that the stain test is of chief importance next to the strain test. Our experience has likewise indicated that a stain test has seemed to be of importance in indicating qualities, not only of the tendency to bleed, but also of such matters as adhesion and skinning. Our experience has been gained merely through the use of pats in metal rings on filter paper resting on a glass plate, somewhat after the manner of some methods that have been publicized. Also a slump test using a metal trough has been used with some satisfaction.

It is realized of course that such procedures do not simulate actual service conditions, but they seem to have some merit, not only in cutting down variables, but also in being more applicable to uses where the material is employed not alone in stone pointing, but in glazing on metal sash. Moreover, such procedures give rather rapid indications which are often of more practical importance than a truer estimate under conditions that approximate actual use more closely. For example, such methods might be useful in eliminating unsuitable compounds. It is this I have in mind in suggesting tests of an accelerated nature.

Mr. H. S. Brightly[2] (*by letter*).—The former Indiana Limestone Quarrymen's Assn. instituted a series of tests on caulking compounds at the Bureau of Standards nearly 10 yr. ago, prompted by the fact that a great many such products in use at that time were not giving satisfactory results. The extensive use of caulking compounds during the past 10 yr. is no doubt partly due to the fact that the investigation referred to brought out basic faults in a number of the materials and inspired manufacturers to overcome these defects.

With regard to the tests proposed by Mr. Kessler, I am inclined to question the merit of the strain tests in so far as the permanent effective sealing of masonry joints is concerned. The procedure proposed is not new. It apparently is an effective means of demonstrating whether the product has the required adhesive and cohesive qualities to permit the necessary stretch without tearing away from the surfaces of the masonry material on the two sides of joints that it is intended to seal. I should like to point out, however, that in the investigation referred to above, it was shown that products which might show a high value during the early period of the investigation, would, in a reasonably short period of time thereafter, through the evaporation and hardening of their solvent, lose those qualities and after comparatively few years, be less satisfactory than products which did not show up so well in the early stages of the test procedure.

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[2] Secretary-Director, Building Stone Association of Indiana, Inc., Bloomington, Ind.

This, it seems to me, points to the need of a test procedure that will develop the strain qualities over a longer period of time. With this in view, I feel that the strain test proposed must be supplemented by some other test that will determine the relative effectiveness of the materials after a reasonably long period of exposure to weather.

MR. D. W. KESSLER[1] (*author's closure by letter*).—The suggestion of Mr. Moore in regard to shortening the test period by means of accelerated curing conditions is a good one. Experiments are being made to determine the effects of various curing conditions such as exposing joints to temperature and moisture cycles, also exposure to carbon arc lamp rays.

Where joints are exposed to a temperature cycle (20 to 120 F.) with intermittent soaking, the caulking usually shrinks and loses plasticity more rapidly than when the joints are kept dry at room temperatures. The carbon arc light as used in the weatherometer test apparently has some action on caulking materials but this does not seem to be as promising for an accelerated test as temperature and moisture cycles.

For the past four years we have been placing the specimens used for slump and staining tests out in the weather for observation. These exposures have given some valuable comparisons between the ordinary test results and the behavior of the materials out of doors. Observations indicate that if a caulking material passes the four routine tests outlined it may be expected to have good weather resisting qualities.

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[1] **Chief, Stone Section, National Bureau of Standards, Washington, D. C.**

THE INFLUENCE OF ENGINE DESIGN ON OIL CONSUMPTION

By M. O. Teetor[1]

Synopsis

Many elements of engine design affect oil consumption. Most of the elements are known but the extent of their individual or combined influence is not very well known. This paper discusses the following subjects and their relation to the control of oil consumption: Oil leaks, cylinder lubrication, theory of oil film action on cylinder walls, piston and ring designs, cylinder distortion, temperatures and finish.

The average automobile owner would define oil consumption as the amount of oil *not* in the crankcase that at some previous time *was* in the crankcase. This definition may be a little broad, but from the owner's viewpoint it is not unreasonable. The car owner is not particularly interested in what happens to the oil. His only interest is in having an engine that does not require "oil attention,"—either adding oil between changes or changing oil too frequently. It is, therefore, not unreasonable to assume that engine design must be refined so that performance in terms of oil consumption will be satisfactory and in accordance with the car owner's definition.

Oil consumption is excessive in an engine only when there is a similar engine in existence that will operate satisfactorily on less oil. Two hundred miles to the gallon at any speed was not unusual at one time. Two thousand miles to the gallon at 60 m.p.h. is not uncommon today. The question, "How much oil should an engine use," has not been definitely answered. There are many factors to be considered in even venturing an opinion on this subject. The opinions expressed on the subject are usually based on past performance and experience which makes them either biased or prejudiced depending upon the factors or combination of factors that have been used as a basis. This paper can only discuss some of the reasons why some automobile engines use more oil than others and stimulate thought and interest in engine designs that have possibilities of operating efficiently with less oil consumption.

Where Does the Oil Go?

The oil in an automobile engine has three ways to escape. It can leak out where shafts project through the crankcase or because of faulty gaskets or packing; it can be drawn into the combustion chambers past the intake valve guides; or it can pass the pistons into the combustion chambers.

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The amount of oil that leaks out of automobiles is tremendous. This is clearly demonstrated by the discoloration of our concrete highways. Main bearings that do not provide for some type of efficient drainage back into the oil pan allow the oil to follow the crankshaft to the outside of the crankcase. The problem of holding oil in a crankcase is not simple but it can be solved—for some engines do not leak oil. The best of gaskets can leak oil because of faulty installation, but when they are properly designed and installed they usually do not leak.

If the correct amount of oil is delivered to the valve alley, the vacuum in the intake manifold will deliver the oil to the valve stems for sufficient lubrication. Whether the valves stick, wear excessively or allow too much oil to escape into the combustion chamber depends upon the design and fitting of the valve guides and stems. Some designs give good service and do not consume excessive amounts of oil which is sufficient proof that this problem is not too difficult.

Although it is recognized that oil can leak out of the crankcase, and that it does pass the intake valve guides, the most attention given the subject of oil consumption has been centered on the amount of oil passing the pistons into the combustion chambers. Oil lost on the road does not cause trouble. It just costs money. Oil that passes the intake-valve guides causes little or no trouble but costs money. Excessive amounts of oil passing the pistons cause plenty of trouble and cost plenty of money in terms of oil, maintenance and the life of the engine.

CYLINDER LUBRICATION

The cylinder wall of an internal combustion engine is one of the few bearing surfaces that can be over-lubricated. The cylinders of an engine are adequately lubricated when a film of oil is maintained on the cylinder walls just thick enough to prevent metal to metal contact. A thicker film will not lubricate more effectively and will result in excessive oil consumption. A thinner film will cause the scuffing of rings, pistons and cylinders.

The terms "scuffing" and "scoring" are used to describe the surface destruction of cylinders, pistons and piston rings. There is a slight distinction between the meaning of these terms. "Scuff" when used in this field usually carries with it a meaning of minor seriousness in contrast with score. A scuffed cylinder, piston and ring assembly can be operated with a fair possibility of its smoothing itself up under normal operation, and improving in its ability to control oil consumption. A scored assembly can be considered beyond redemption as far as improving in oil consumption or smoothing up under operation. A scored assembly has undoubtedly at some previous time been only slightly scuffed, but all scuffing does not necessarily develop into scoring. The type of scuffing that does not develop into scoring is one of the important subjects in this paper because oil consumption cannot be maintained at an economical point or controlled when the bearing faces of the rings and cylinders are scuffed. Insufficient lubrication can probably be conceded to be the basic reason for all scuffing, but lubricating oil has its limitations and engine design must always compensate for these limitations.

Oil Delivery to the Cylinders.—The conventional system of oil delivery to a cylinder is through a squirt hole in the crank end of the connecting rod. Although this system is in general use, the size hole used in the connecting rod and the indexing of this hole with the delivery hole in the crankpin vary considerably with different engines. If the oiling system is properly designed, the volume of oil thrown into the cylinders for adequate cylinder lubrication is not too great to be economically controlled by cylinder, piston and ring design.

ACTION OF THICK AND THIN FILMS OF OIL ON CYLINDER WALLS

The oil film maintained between the cylinder wall and the compression rings, and between the rings and the ring grooves in the piston, seals the engine against blow-by. A thick film of oil between these surfaces would be ideal for sealing the engine against blow-by, but for other reasons a thick film is not desirable. A piston must have clearance in a cylinder for an oil film plus additional clearance for thermal adjustment. A piston ring must have edge clearance to allow it to expand and contract freely to follow the cylinder wall. The necessary clearance between these surfaces is more than ample for a thick film of oil, but a thick film is difficult to control and a thin film is sufficient for adequate lubrication. The pressures between these surfaces are varied and constantly changing so that the oil is constantly on the move. The trend of the movement is into the combustion chamber where the oil not only ceases to be useful but becomes troublesome.

It is significant that when used oil is clean the oil consumption of the engine is usually low and wear is almost insignificant. When used oil is dirty, oil consumption is usually high accompanied by excessive wear. The following explanation for this phenomenon seems reasonable. The oil film on a cylinder wall is exposed to the heat of the combustion chamber when the piston is at the bottom of the power stroke. Some of the oil on the cylinder wall will be burned each power stroke. The amount burned depends largely upon the thickness of the oil film. If the film is thin the heat can readily pass through the oil into the cylinder wall and not destroy the oil.¶A thick film of oil obstructs the heat flow and the surface of the oil exposed to the heat burns to carbon. The carbon thus formed mixes with the oil on the cylinder wall and some of it is constantly finding its way back into the crankcase past the rings. For this reason an engine consuming large quantities of oil is probably operating with a thick film of oil on the cylinder walls which is being attacked by the heat and turned into carbon which changes the color of the oil. The more oil an engine uses the more contaminated the oil becomes. There is such a definite relation between the color of used oil and oil consumption when the other variables affecting oil consumption are held constant that the oil consumption can be closely estimated by the color of the used oil. Contaminated oil is not as good a lubricant as clean oil which is therefore a logical explanation for the relationship between excessive oil consumption and excessive wear.

A thin film of oil is sufficient for adequate lubrication as long as the film is not broken. There is less chance of it being burned by the combustion heat if the thickness is uniform and sufficiently thin to allow the heat to pass readily into the cylinder wall. It is probably not known just how thin this oil film could be, but an idea of about how thin it is in an economical engine can be ascertained from the following figures: The oil film on the cylinder wall of an 8-cylinder engine that is running 2000 miles to the gallon is so thin that it will hardly show when wiped off with a white cloth. The oil consumed per cylinder per stroke in such an engine would be 0.000005 cu. in. If a uniform layer of oil is burned from the flame-swept area of the cylinder barrel, the oil film consumed would be only 0.0000001 in. thick. If the oil film retained on the cylinder wall plus the film consumed is hardly detectable, the film necessary for adequate lubrication is probably microscopic in thickness.

PISTON INFLUENCE ON OIL CONSUMPTION

The bottom of the piston skirt performs the first thinning operation on the oil film. Its efficiency in cutting down the thickness of the oil film is largely determined by its clearance in the cylinder and its ability to contact the cylinder wall uniformly around its entire circumference. Pistons that have the lower outside corner beveled or rounded do not assist in keeping the oil film thin because they ride over the oil film rather than help scrape it off. When the piston skirt does not scrape the oil film off of the cylinder wall to some extent, the entire oil control falls upon the piston rings. Piston rings can be designed to control only a certain volume of oil without other disastrous effects so the assistance of the piston skirt is important.

Piston-skirt pressures must be sufficiently uniform under all operating conditions not to break through the oil film and produce dry streaks in the cylinders. Concentrated spotty skirt pressures are usually caused by piston skirt distortion. All pistons have some distortion at some speeds. Some pistons do not have sufficient distortion within the speed range at which they are operated to break through thin oil films and are therefore very satisfactory. Whenever a piston skirt has sufficient distortion to break through an oil film, the results are disastrous to the life and performance of the engine because the skirt of a piston will scuff, and scuff the cylinder. The surfaces of the piston rings will scuff when they pass over the dry scuffed streak in the cylinder. The rings turn circumferentially on the pistons. As they rotate they present a new section of the surface each stroke to the scuffed streak in the cylinder which will destroy the entire surface of the rings in a very short time.

A piston is a combination ring carrier and cross-head. The piston skirt must present sufficient rigid bearing surface to the cylinder to ride straight up and down in the cylinder. The piston head carrying the rings must be supported by the skirt of the piston. If the piston skirt is not sufficiently rigid to resist collapse or operates with excessive clearance, the head or lands between the rings can strike the cylinder wall. When the head or lands between the rings strike the cylinder wall they either disturb or break through the oil film. When the rings pass over these spots where the oil film is broken, they start a scuffing action which will continue as long as the piston head strikes the cylinder wall. Any movement from side to side or rocking action about the piston pin hampers the operation of the piston rings. If piston movement is too great it will rock the rings off the cylinder wall and break the oil seal between the faces of the rings and the cylinder. Continued operation under this condition will wear the faces of the rings oval. Oval-faced rings will slide over the oil and therefore cannot be efficient in maintaining a thin oil film. A piston ring's effectiveness in controlling oil consumption depends to a great extent upon its ability to maintain sharp uniform shearing edges in contact with the cylinder wall.

CYLINDER DISTORTION

Cylinders that are accurately machined straight and round at room temperature do not stay straight and round after the engine is up to temperature and in operation. Mechanical and thermal distortion changes the shape of the cylinder barrels under operating conditions. Mechanical distortion of cylinders that affects oil consumption is caused by studs and bolts in the cylinder walls or in adjacent sections that hold other parts of the engine to the cylinder block. Of the entire classification, cylinder-head studs are the worst offenders.

In order to get a clear conception of the amount of cylinder distortion in our present-day automobile engines, the cylinders of a new engine should be examined after about five or six hours' running. At the end of this short run the piston rings have had an opportunity to show up the particular parts of the barrels that do not conform to their accurate shape. Any high spots or protruding areas will be polished by the rings. The areas that are pulled away from the round contour of the cylinder will appear light brown in color because the rings will not have had contact or sufficient pressure in these areas to scrape the burned oil off the surface. The irregularities in the cylinder surface show up very plainly after a few hours of running, but after this first development period the piston rings start the process of wearing the cylinder round while it is in its heated, distorted condition.

Figure 1 is a photograph of a typical example of mechanical distortion.

It shows a pocket pulled in the top of a cylinder barrel which has been caused by the tension on a cylinder-head stud. The design of the stud anchorage or its location is responsible. The variation of the contour of a cylinder surface from a true circle caused by mechanical distortion is frequently as much as 0.002 or 0.003 in., and in some cases has been found to be as much as 0.005 in. The seriousness of this situation is increased by the fact that the area of such distorted portions is often very small. The depressed or protruding areas are frequently in the form of spots or streaks similar to the distortion shown in Fig. 1. This type of distortion forms a series of humps and depressions throughout the entire circumference of the cylinder to which neither pistons nor piston rings can even reasonably conform.

The improvement in oil consumption that can be effected by the elimination of most of the mechanical distortion can be demonstrated by finish honing cylinders with the head bolted in position. This operation can be done by using a special head provided with holes through which the hone can be operated. The cylinders are then finished with the tension on the cylinder head studs which is the factor that distorts the top of the cylinder barrels. A special hydraulic torque-indicating wrench is used to tighten the cylinder head nuts so that the tension can be duplicated. When cylinders are finish honed in this manner the mechanical distortion of the cylinder barrels is reduced to a minimum. Comparative oil-consumption tests have been made on many engines with the cylinders finished conventionally and then finished with a special head. The elimination of most of the mechanical distortion by this special finishing process has improved the performance of engines in terms of oil consumption as much as 500 per cent. Cylinders comparatively free from mechanical distortion are so much more economical in the consumption of oil that in

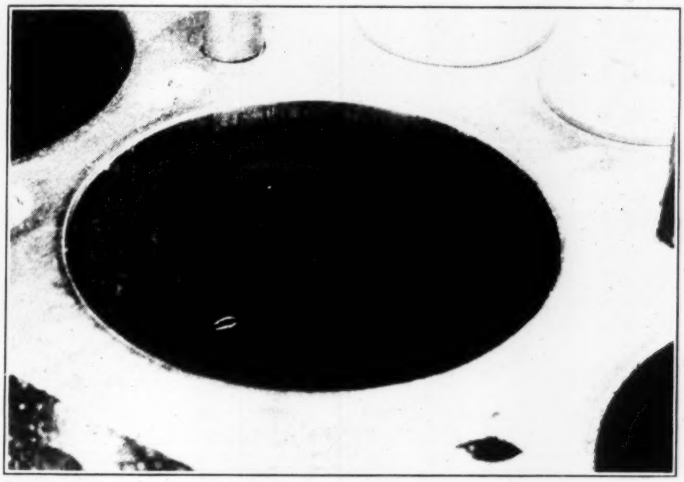

FIG. 1.—Typical Example of Mechanical Distortion.

some instances engines have actually been built in production with the cylinders finished with special heads bolted in place.

Thermal distortion of cylinder barrels is distortion caused by some portions operating at higher temperatures than other portions. The fundamental principle upon which an internal combustion engine operates generates the heat in the combustion chambers and in the upper zone of the cylinder barrels. If the sections of the cylinder block in direct contact with the heat are allowed to get very much hotter than other sections they will expand and distort the cylinder barrels in some manner.

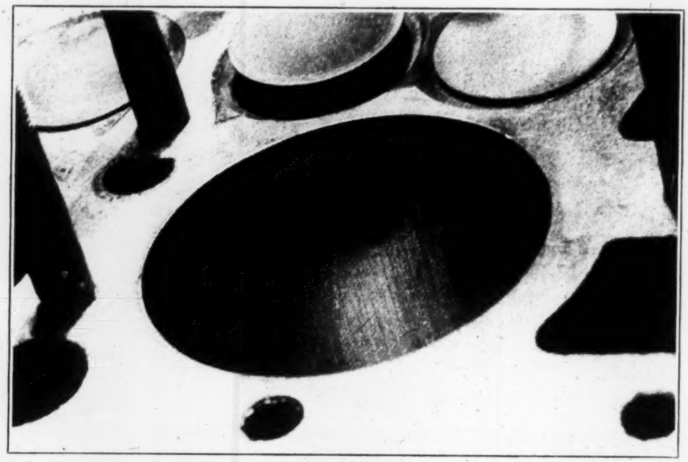

FIG. 2.—Showing a Spot in a Cylinder Wall that Has Expanded Away from the Round Contour of the Cylinder.

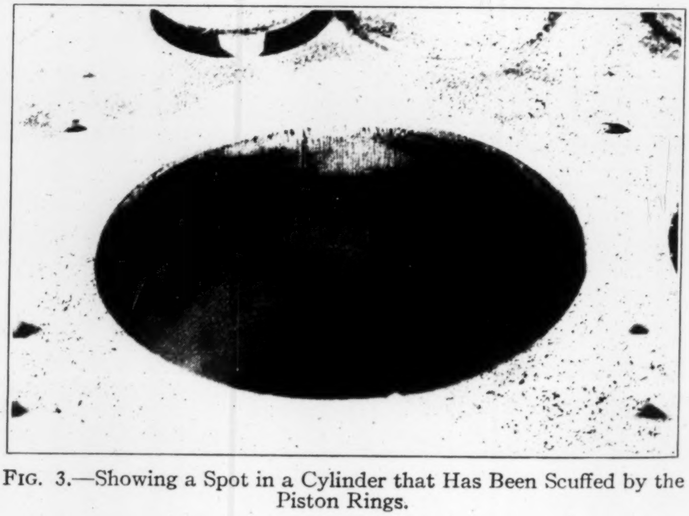

FIG. 3.—Showing a Spot in a Cylinder that Has Been Scuffed by the Piston Rings.

Figure 2 is a typical illustration of a spot in a cylinder wall that has expanded away from the round contour of the cylinder because of high temperature. Water jackets were originally designed with the idea in mind that as long as the hotter portions of the cylinder blocks were surrounded by water they would cool sufficiently by thermosiphon or by a slight circulation of the water. If the engine would not visibly over-heat the cooling system was adequate. The many improvements that have been made in engine design have made it possible to produce more power with the same size or smaller engines. When more power is produced more heat is generated and the cooling system that was once adequate becomes insufficient. The additional heat generated is localized in spots which increases the differential in temperature between the different portions of the cylinder barrel. As the spread in temperature increases, thermal distortion increases. Cooling systems must therefore be designed to direct the coldest water into the hottest spots in sufficient volume to reduce the temperature differential.

CYLINDER-WALL TEMPERATURES

Hot spots in cylinder barrels do double damage. Not only do the cylinders warp out of shape because of thermal distortion so that the piston rings cannot control the thickness of the oil film but some of these spots get so hot that they cannot be lubricated. Figure 3 shows a spot in a cylinder that has been scuffed by the piston rings. This particular area in this type of engine is subjected to the blow-torch action of the combustion chamber flame as it passes over the top of the cylinder to the exhaust valve. The restricted space provided for water between the cylinder barrel and the exhaust valve, or the insufficient volume of water passing through this space, is responsible for the high temperature at this spot which has been high enough to destroy lubrication. It is not uncommon to find temperatures as high as 650 F. in this location.

Several fundamental factors in the design of an L-head engine make this spot one of the most difficult to cool. Space must be provided for cooling water between the cylinder barrel and the valve ports and yet at the same time it is advantageous to have the valves as close to the cylinder barrel as possible. A compromise in design between adequate space for water and a more efficient combustion-chamber shape is what seems to cause the trouble. If space for cooling water is sacrificed in order to move the valves a little closer to the cylinder the engine will probably produce more power which in the end will generate more heat to be absorbed less efficiently because of less cooling water. The heat is regenerative in another way because steam forms in these narrow passages and lime deposits form, which in a very short time will entirely close the passage and eliminate the cooling entirely.

Some engines when operated at top speed or full load or even sometimes within 80 per cent of top speed or full load will develop hot spots in the cylinder barrels that are too hot to be lubricated. The oil on the surface of a hot spot in the cylinder decreases in viscosity because of the high temperature to the point where the strength of the oil film is not sufficient to withstand the pressure of the piston rings. The hot blow-by gas in the combustion chamber passes between the ring and the cylinder wall generating more heat because the oil seal has been broken. Therefore starting with the breaking down of the lubrication the temperature of the hot spot increases rapidly and scuffs both the high-temperature area in the cylinder barrel and the surfaces of the piston rings.

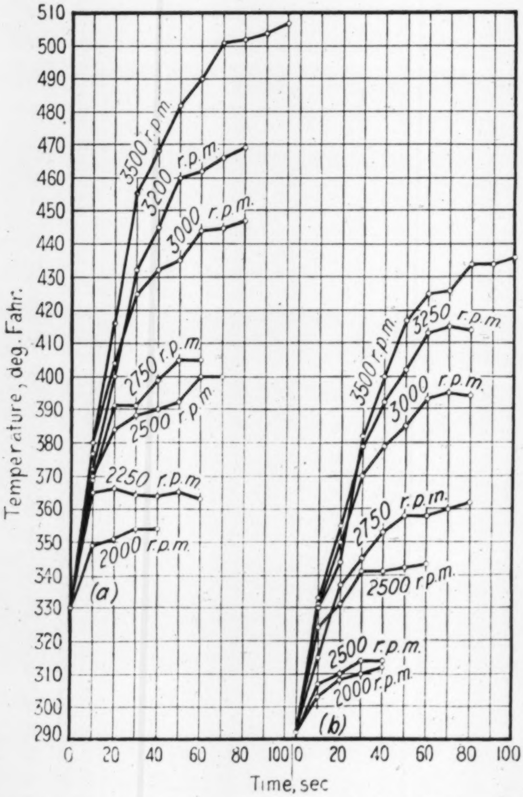

FIG. 4.—Cylinder Wall Hot-Spot Temperature Rise in Relation to Engine Speed at Road Load.

(*a*) The original temperature was taken at 1500 r.p.m. without load and accelerated to speed before the load was applied. Timing started with the opening of the throttle.

(*b*) Temperatures taken after lime and core sand had been removed from the water jacket between the valve ports and cylinder barrel following the same test procedure as (*a*).

Accurate cylinder wall temperatures are rather difficult to obtain. Average cylinder wall section temperatures useful in designing efficient cooling systems can be obtained by brazing thermocouples in the cylinder wall sections with the ends of the wires finished flush with the cylinder surface. The water temperature influence can be eliminated by insulating the thermocouple wires through the water jacket to the outside of the block. Cylinder surface temperatures can be recorded in the same manner by a slight change in the mounting of the thermocouple. In order to check surface temperatures it is necessary to stop some of the heat flow from the thermocouple to the cylinder wall. This can be accomplished to some degree by mounting the thermocouple in the center of a taper plug approximately $\frac{1}{16}$ in. thick. The taper plug is then driven into a reamed hole and finished to the contour of the cylinder wall. The water is packed off on the water jacket side and the wires insulated to the outside of the cylinder block. Probably neither the sectional temperatures nor the surface temperatures recorded in this manner are correct but at least they are on the low side. Since the temperatures recorded in this manner are sufficiently high to cause concern, the extreme accuracy of the temperatures becomes rather unimportant.

The temperatures of cylinder wall hot spots go up and down with the throttle opening. The time required for a hot spot to reach maximum temperature at a definite speed and load depends to a certain extent upon the efficiency of the cooling system. Figure 4 shows temperature rise in relation to engine speed and the time in seconds required for the cylinder wall to reach a maximum temperature at a given speed. The thermocouple used in this test was located in the cylinder wall midway between the valves and in the middle of the top dead-center position of the top piston ring. The cylinder used in this test was conventionally designed with respect to cooling water capacity and circulation. The temperatures indicated in Fig. 4 (*a*) were sufficiently high to scuff the piston rings. After these temperatures were taken the cylinder was examined for obstructions in the water jacket and a small quantity of core sand and lime was removed from the space between the cylinder wall and the valve ports. Figure 4 (*b*) shows cylinder wall temperatures after the water jacket was cleaned out. The decrease in cylinder wall temperature eliminated almost all the scuffing of the piston rings which indicates that cylinder lubrication starts to break down after the wall has reached a temperature of approximately 400 F.

Figure 5 indicates the position of ten thermocouples located in the cylinder wall and roof of a single cylinder block used on an experimental test engine. The cylinder used for this work was conventionally designed with respect to the water circulation but the water passages were larger than those ordinarily used. The highest temperatures recorded are therefore considerably lower than will be found in some of our present-day engines. Figure 6 is a record of the temperatures recorded at the different thermocouple locations from 2000 to 3500 r.p.m. full load. The highest cylinder

Geo. C. Smith

wall temperature recorded was 292 F. The hottest area in this cylinder wall was in the top of the barrel on the valve side as indicated by thermocouples 7 and 8. These points were considerably hotter than thermocouple 4 which was only ½ in. below 7 which indicates how small these hot areas can be. When the differential in temperature is great between a small hot area and the surrounding surface the hot area expands and distorts the cylinder surface producing small irregularities in the surface. When the piston rings pass over these areas the ring pressure is concentrated on the small protruding spots. The viscosity of the oil on these hot areas is very

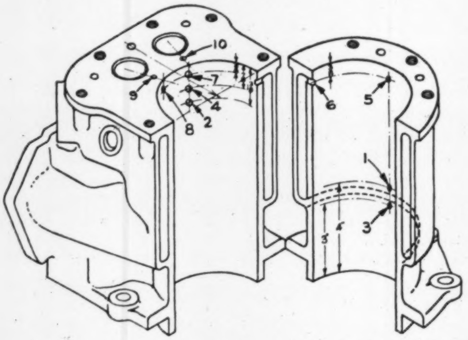

FIG. 5.—Locations of Thermocouples in Single-Cylinder Test Engine.

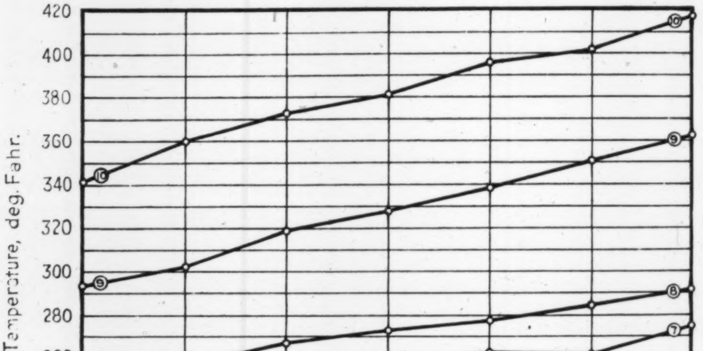

FIG. 6.—Cylinder Wall Temperatures Indicated by Readings Taken at Ten Different Locations at Speeds from 2000 to 3500 r.p.m.

low. The combination of increased pressure and decreased oil-film strength results in scuffing. Thermocouples 9 and 10 in the roof between the valves and the cylinder barrel indicate much higher temperatures than found on the cylinder wall. The expansion of this section distorts the top of the cylinder barrel.

There is a differential in temperature between thermocouples 1 and 3. This particular test shows a difference of only 10 to 15 deg. between these two points but in cylinders of similar design with less efficient cooling systems this differential is much greater. This temperature differential seems rather unimportant but in this particular location it has a very great influence on oil consumption. The temperatures above and below where the water jacket joins the cylinder barrel are quite consistent around the entire circumference of the cylinder. Under operation, these two temperatures establish two different cylinder diameters very close together vertically. When the piston rings ride over the junction of these two different diameters they actually collapse and leave the cylinder wall. An engine of this particular design will not be economical in the consumption of oil until the piston rings have actually straightened the cylinder barrel so that it is straight when the cylinder block is up to operating temperatures. A piston reciprocating in a cylinder with the rings rotating on the head makes a rather efficient tool for producing a round straight hole. This attempt at making a round straight hole in which to operate is the life long job of pistons and rings when they operate in cylinders that are either mechanically or thermally distorted. The piston rings therefore actually attempt to wear a cylinder straight and round when the cylinder block is up to operating temperatures and with considerable success.

One engine having the junction of the water jacket in the piston ring travel continually improved in oil consumption throughout a 60,000-mile dynamometer run. When the test started, the first checks showed oil consumption at the rate of 600 miles per gal. at 60 m.p.h. After 60,000 miles the oil mileage had increased to 2500 miles per gal. Before the test started, the cylinder barrels were carefully measured and they checked commercially round and straight. The cylinders were measured after the test and the diameter at the junction of the water jacket and cylinder barrel had increased 0.0025 in. with an increase of only 0.001 in. on each side of the junction.

Hot spots in cylinder walls have been eliminated to a great extent in some of the recently designed cylinder blocks by the use of larger water passages and circulating systems that direct the cold water into the hot areas. The temperature in the hottest spot of one of these new engines varies only 20 deg. Fahr. from 2600 to 4200 r.p.m. full load. The elimination of hot spots in cylinders increases the possibility of adequately lubricating them at higher speeds and loads. Cylinder barrels that are approximately uniform in temperature throughout their entire surface under operating conditions stay more nearly straight and round. Straight round cylinders give the piston rings an opportunity to function properly which opens up new possibilities for minimum oil consumption and longer engine life.

CYLINDER FINISH

Cylinder finish is directly related to oil consumption because of its influence upon initial performance. Cylinder surfaces wear smooth unless influenced by unusual operating conditions. If cylinders are to wear to a certain smoothness it is only reasonable to assume that cylinders should be finished in the first place at least as smooth as the resultant worn surface. In saving the amount of wear necessary to smooth a rough cylinder, many thousands of miles are added to the life of the piston rings because the piston rings are primarily responsible for the worn finish of a cylinder. If a rough cylinder wears to a polish, the performance so far as oil con-consumption is concerned may be very good but the cast iron worn off the faces of the rings and cylinder wall when mixed with the oil will prematurely wear out the entire engine. If the cylinders are rough and the initial run-in conditions are such that the rings start scuffing, engine life is impaired and oil consumption cannot be controlled.

WHAT CAN BE EXPECTED OF PISTON RINGS?

With all of these factors affecting oil consumption and the maintenance of adequate lubrication it looks almost impossible to be able to control either oil consumption or lubrication with piston rings alone. This statement is absolutely true with some engines. Some of these other factors, or combinations of these other factors, are so unfavorable to adequate lubrication and oil control that the performance of some engines in terms of oil consumption is consistently bad regardless of the character of the piston rings used. Correctly designed piston rings perform better in almost all engines than poorly designed rings in spite of all the factors affecting their performance. Piston rings must have the proper shape and pressure characteristics to follow the cylinder wall and oppose as many as possible of the influences tending to make them leave the wall. Of course it is reasonable to assume that enough pressure could be applied to force them to stay on the wall but oil films still have their strength limitations and increased wall pressure to such a figure would mean an increase in friction.

The reasonable solution seems to lie in improvements in engine design that will eliminate the unfavorable factors affecting the performance of piston rings. Considerable progress has already been made in this direction proving that some of these unfavorable factors can be eliminated. It is not at all unreasonable to hope for the not far-distant day when the lubricating oil in the engine will require no more attention than the lubrication of the differential and transmission.

DISCUSSION

MR. S. W. SPARROW[1] (*presented in written form*).—The author has covered his subject accurately and completely and the information with respect to cylinder wall temperatures is a particularly welcome addition to our knowledge of internal combustion engines. Comments which follow merely amplify one or two of the items which he has mentioned.

With reference to cylinder lubrication it is stated that "The conventional system of oil delivery to a cylinder is through a squirt hole in the crank end of the connecting rod." I believe that ordinarily this hole is designed to supply lubricant to prevent scuffing during cold starting or to prevent piston slap during slow speed operation. The author, I think will agree that, under most conditions of engine operation, the cylinder walls receive sufficient oil from the ends of the connecting rod bearings and from the crank cheeks.

As for scuffing, it should be noted that this is attributed to insufficient *lubrication* and not to insufficient *lubricant*. This is an important distinction for while the scuffing during the starting period may be due to a lack of oil, it is very unusual to find that scuffing or scoring at high speeds can be corrected by supplying more oil to the cylinder walls. As the author explains, lubrication fails because of *high* spots or *hot* spots. The high spots may be a consequence of distortion or surface roughness, and metal to metal contact results because the unit pressures are abnormally high. At the hot spots, contact occurs because the viscosity of the oil is abnormally low.

Scuffing may be initiated by a compression ring or a piston land at the top of the bore where the temperatures are exceedingly high. More often it starts from the oil control ring which, although more favorably situated as regards temperature conditions, must exert high unit pressures if it is to maintain the much desired thin oil film. This ring is not unlike a razor blade which must give a complete shave with a single stroke. For good oil economy it must give a "close shave" and this is disastrous to stray "pimples" caused by distortion and to the "tender spots" which are due to high temperatures. Moreover, rocking of the piston is like the trembling of the hand that holds the razor. Nevertheless, thanks to organizations such as Mr. Teetor represents, reasonable oil economy is obtainable from engines that are still far from perfect.

MR. M. M. ROENSCH.[2]—A very few years ago, a car running 800 miles per gal., at 60 m.p.h., was very satisfactory, and one of the main reasons was the fact that very few cars were driven at 60 m.p.h., most people drove 40 to 50 m.p.h. and therefore had good economy at that speed.

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[1] Mechanical Engineer, Studebaker Corp., South Bend, Ind.

[2] Research Engineer, Research Laboratory, Chrysler Corp., Detroit, Mich.

Road speed has now increased to 60 to 70 m.p.h. and the car owner that does not get about 2000 miles per gal. is dissatisfied. To meet this condition, both the ring manufacturers and the engine designers have been put to it to get this good economy and to find, at the same time, the magnitude of factors affecting economy, so as to be able to have the future work mean something.

A number of people are of the belief that too little oil consumption can be harmful to the engine, and excessive wear results will occur if we do not use some oil. Wear occurs from two causes, (1) dirt, (2) high spots and hot spots in cylinders. We, in our laboratory and road tests, have been unable, up to the present time, even with oil consumption as low as 10,000 miles per gal., to find any indications of excessive wear due to good oil economy. In fact, in looking over our tests for the last few years, the wear per 1000 miles of operation is decreasing. It is our desire in our engines, to get the oil consumption to a point where it is less sensitive to oil viscosity. The oil companies are endeavoring also to flatten the viscosity curve, which is in the same direction.

At the present time, owners are using a more viscous oil during the summer, mainly because with the light oil, it is difficult to control oil consumption at the high crankcase temperature encountered in hot weather. I believe it is entirely possible not only to design the engine to control the lighter oil from the economy standpoint, but also the engine can be designed so that the crankcase temperature will not be excessive, and by reducing the crankcase oil temperature at high speeds 50 deg. or more, a lighter oil can be used with equal satisfaction.

Mr. Teetor's paper has mentioned many of the factors of design that must be considered to make this low oil consumption possible, and in my experience, the most important single factor in the engine design is that the cylinder barrel be so designed that it will remain cylindrical throughout the entire operating range of speeds and loads.

Several years ago we used to wish for some means to find the shape of the cylinder barrel under actual operating conditions. Finally, through careful measuring of cylinders before and after certain tests, we began to see the handwriting on the cylinder wall. We now find the shape of the cylinder barrel under operating conditions by starting with a straight, round barrel and running the engine under fixed loads and speeds for the equivalent of several thousand miles and then remeasuring the cylinder.

The piston rings that control the oil and seal the compression have performed another service, they have machined the cylinder to its hot shape and so when the cylinder is cold, the places that were high points under operating conditions, are now low, and show up as such in our diagrams. The elimination of the thermal distortion of the cylinder under operating conditions remains still the most difficult problem.

Mr. Teetor has shown very clearly the way the distortion from cylinder head studs and the like can be limited, but to so cool the cylinder barrel so that we shall not have a temperature difference of 300 to 400 deg. between the valve side and the opposite side of the cylinder remains today our most difficult problem, though a 200 to 300 per cent improvement has been made in this direction in the past few years.

MR. ALEX TAUB.[1]—There are many factors that affect oil consumption in automobile engines. Leaks, of course, must be considered, but I do not think that leaks are as important as they were a couple of years ago.

Straight bores, of course, are necessary, but looking over a large number of comparative and competitive cars, it is gratifying to see how well the cylinder bore job is being done today. There is a vast difference between this year and two years ago. The bores are coming straight to 0.0005 in. today where three years ago they were still working in thousandths, so I think that the straight bore is definitely with us.

The question of piston taper is important. If you have a reverse taper, by that I mean smaller on the bottom of the skirt than at the top of the skirt, a matter of 0.0015 in., you can lose 50 per cent in oil consumption. In other words, your oil consumption will go up 100 per cent, so reverse taper on pistons is important; and if anything, pistons should be tapered larger on the bottom than on the top.

Uniform circumferential fit in the block—you do not get it anyway. The majority of pistons are not round; they are machined or ground oval. In the aluminum piston you have the additional factor to overcome, that of piston breathing. Any piston that is structurally weak will breathe, and this breathing has a habit of upsetting the ring contact with the bores and interfering with oil consumption.

Reference has been made to bore distortion at the top of the block. But there is another distortion equally bad that we are inclined to ignore—the distortion in the bottom of the block. It does not show up in black marks on the bores; it is a black mark, at least against the engineering department, if not minimized.

In spite of what we may do, crankcases under high speeds move, particularly in the center. The tendency is for the crankcase to swing like a bell at the center, being fairly stationary at each end. The crankcase cannot move very far without taking the cylinder barrels with it. It is necessary to analyze these movements and so construct the roof of the crankcase, where the barrel intersects the crankcase, to be sure that "bending" of the barrel is minimized, and that the point about which movement is centered is not in the middle of the barrel. It must be worked out so that the tendency to move, if any, is centered at the top; then if it does move, it is in a fairly straight line.

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[1] Power Plant Development Engineer, Chevrolet Motor Car Co., Detroit, Mich.

There are jobs that move as much as 0.014 in. across the center, the average barrel moving 0.008 to 0.009 in. across the center. You can draw your own conclusions as to what happens to the rings in the center cylinders when they move. This is a problem in crankcase design.

As far as wear is concerned in the cylinder barrel, tin plating has done a rather marvelous, if unlooked-for, job. Tin plating was put on pistons to permit more abusive handling and to take care of the variations in shape that do occur, even in a good block. With the introduction of tin plate, we found that wear dropped immediately to one-tenth of what it was originally without any other change, and very little tin is taken off the piston. We have a series of cars, that have just completed their regular run, and instead of having a normal 0.0025 to 0.003-in. wear, we have 0.0002 and 0.0003 in.

As for temperature affecting oil consumption, naturally there is some effect as the temperature goes up, but it does not compare with the effect on the initial viscosity of the oil. We are going to design engines to give satisfactory oil consumption with the lighter oils. We hope that the ring-makers will do their part, so that we can use the light oils the year around.

Progress is still to be made through greater accuracy and better control in the manufacture of rings. Particularly is this important in the matter of wall pressure. The difference between a 9-lb. and 12-lb. oil control ring may be 50 per cent in oil consumption. If I were asked to say what we need, I should say we need rings that break in quicker. We need rings that have a minimum change in the consumption over a long period of miles, and we want the maximum wall pressure and the minimum wear and the maximum seal. The drains in these oil rings should not plug. They may plug—it would be nice if they did not; drilled rings are somewhat better in this respect.

In closing, I should like to say, also, that we have heard a lot about the mirror-finished surfaces. In our experience, we do not see where that serves any useful purpose. It certainly does not help the job of running in. It does not help distortion, and if you get a job that is really round, not subject to mechanical distortion or thermal distortion, you will not need a mirror finish.

MR. R. R. TEETOR.[1]—I was very much interested in Mr. Sparrow's comments and was glad to know that he was in complete agreement with the experience we have had.

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[1] In Charge of Engineering, The Perfect Circle Co., Hagerstown, Ind.

Referring to Mr. Roensch's discussion, I should like to say that we heartily agree with him in his statement that good oil economy is usually a protection against engine wear. We have never seen a single case in which oil economy has been sufficiently great to induce wear in cylinders, pistons or rings, and in every case we have ever studied, the greater the economy the finer the condition of the engine after long service. We therefore believe that maximum oil economy promotes maximum efficiency and greatest engine life.

We wish to thank the Society for the opportunity to present these factors that we have found in our various investigations. I can assure Mr. Taub that when we know enough to make better piston rings we will make them.

THE EFFECT OF VOLATILITY ON OIL CONSUMPTION

By L. L. Davis[1] and R. D. Best[1]

Synopsis

The increasing use of the lighter S.A.E. grades, Nos. 10 and 20, of crankcase oils as compared to the heavier grades and the inherent greater consumption of the lighter grades makes it desirable to determine the factors controlling consumption. Previous work, in general, has indicated that viscosity is the major factor controlling consumption and that volatility is of little or no importance. The present work attempts to isolate the effects of volatility and viscosity. A series of S.A.E. No. 30 oils were prepared from the same crude having similar viscosity but widely different volatility characteristics. These oils were tested for relative consumption on a 6-cylinder engine-dynamometer assembly at speeds and temperature conditions similar to 40, 50 and 60 m.p.h. The results show a wide difference in consumption due to volatility. In the uniform series of oils tested, there appears to be a relationship between the consumption and the front end volatility as measured by the initial boiling point under 2 mm. absolute pressure. This relationship does not hold for oils of widely different viscosity-temperature characteristics, in which case the volatility effect is probably a function of the over-all distillation characteristics. The work indicates that volatility is a more important factor of consumption than has hitherto been thought. It is believed that, at least in the lighter grades of oil, volatility must be considered and that additional work is needed to thoroughly study the separate effects of volatility and viscosity on consumption of crankcase oils.

The recent increase in the consumption of the lighter grades of motor oil as compared to the heavier has resulted in a greatly increased demand for the lower viscosity or so-called "neutral" base oils for blending purposes. This tendency has upset the balance of production of neutral in relation to bright stocks, and if it continues there is the possibility of a definite shortage of the lower-viscosity oils. The available supply of low-viscosity blending oils is limited by the permissible volatility or distillation range. There is, therefore, an economic need for determining the effect of the volatility on consumption in order to conserve the supply of neutral oils.

The effect of the grade of neutral oil used on the available supply of an S.A.E. No. 30 blended oil is shown in Table I. Thus, it is possible to produce 66 per cent of finished 150 viscosity at 100 F. neutral oil from a typical Mid-Continent pressed (dewaxed) paraffin distillate. About 45 per cent of this neutral is required when blended with bright stock to produce an S.A.E. No. 30 motor oil. Therefore, 146 bbl. of the S.A.E.

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[1] Supervisor of Process Division, and Research Engineer, respectively, Continental Oil Co., Ponca City, Okla.

No. 30 oil can be produced from 100 bbl. of original pressed distillate. If, however, the distillate is reduced to a 300 viscosity neutral, only 41 bbl. of S.A.E. No. 30 oil could be produced.

Although a large amount of work has been carried out on the question of motor oil consumption, the net conclusions which can be drawn from the literature are far from satisfactory. In general the investigators have concluded that viscosity is the major factor controlling consumption and that volatility has little or no significance. The majority of the published work has been based upon the examination of a limited group of oils, usually varying over a wide range of viscosity. The resultant consumption data when plotted against viscosity shows a fairly good relationship. It seems to have been overlooked, however, that in such a series of oils there is also a fair correlation between viscosity and volatility, and therefore if the consumption data are plotted against volatility an equally good relationship may result. As far as the authors know, there has been no work published which definitely isolates the effect of the variables of viscosity and volatility.

TABLE I.—EFFECT OF GRADE OF NEUTRAL OIL ON THE AVAILABLE SUPPLY OF S.A.E. NO. 30 BLENDED OIL.

Viscosity at 100 F. of Neutral Oil Used in Blend	Yield of Neutral Oil from Pressed Distillate, per cent	Percentage of Neutral in S.A.E. No. 30 Oil	Barrels of S.A.E. No. 30 Available from 100 bbl. of Pressed Distillate
150	66	45	146
200	43	58	74
240	35	61	57
300	28	69	41

The authors conceived the possibility of isolating the effect of volatility by preparing a series of oils in which that was the only variable. Such a series could be prepared by blending several neutral oils of varying volatility with bright stock from the same crude oil. The finished lubricants would then all be of practically the same viscosity index and would likewise be quite similar in other properties. If the finished lubricants were blended to be equal in viscosity, then the only property which would be significantly different between them would be volatility. This possibility solved the problem of making corrections for variables which were unknown in magnitude.

Another factor which has made it difficult to draw conclusions from past work is that long-time engine tests are practically impossible to check because of changing mechanical and operating conditions. In long-time tests both the volatility and viscosity of the oil may change widely during the test, thus making it impossible to correlate the results from different oils. Stewart and Risk in a paper before the Society of Automotive Engineers[1] described a method of making accurate oil-consumption tests in a short time. The apparatus used in our work was essentially that of Stewart and Risk, but differed in some details. It was built around a 1933 Chevrolet engine. A diagram of the arrangement is shown in Fig. 1.

METHOD OF TEST

The engine was operated with a dry crankcase, the oil being circulated from two outside reservoirs *L* and *A*, the latter being mounted on the scale *B*. Under test operation the oil was drawn from *A* and delivered to the engine by the regular pump *D* through the manually operated pressure by-pass valve *E*. The used oil was circulated by pump *I* from the crankcase through the thermostatically controlled cooler *K* back to the reservoir *A*. As the oil was consumed, the time interval was noted for successive small decrements (usually 0.025 lb.) in the weight on the scale. When it was desired to change the oil under test without stopping the engine, the oil circulation was switched to reservoir *L* and two 6-qt. flushing charges of new oil were circulated to remove the old oil from the engine. Reservoir *A* was cleaned and seven quarts of the new oil added. All oil charged to the system was brought to the operating temperature in the steam coil heated tanks *R* and *P*. After flushing, the engine was switched back to system *A* to test the new oil.

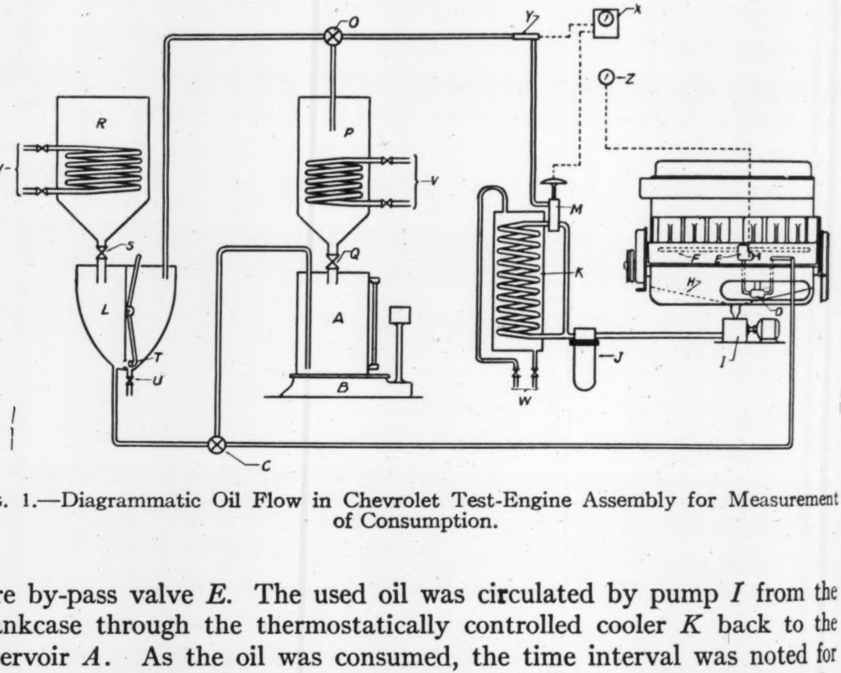

FIG. 1.—Diagrammatic Oil Flow in Chevrolet Test-Engine Assembly for Measurement of Consumption.

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[1] J. P. Stewart and T. H. Risk, "Factors Affecting Oil Consumption," *Journal*, Soc. Automotive Engrs. October, 1933, p. 42.

The engine operating conditions, given below, were selected to match the conditions observed in a fleet of five test cars of the same model, operating during July and August on Oklahoma highways:

	40 m.p.h.	50 m.p.h.	60 m.p.h.
Water temperature, deg. Fahr	180	180	180
Oil sump temperature, deg. Fahr	205	225	235
Gasoline consumption, miles per gal	18.5	18	15
Load, hp	14.1	22.4	34.8
Oil circulated, gal. per min	1.67	2.0	2.11

By controlling the oil pressure at the same values as observed in the field and by the induced ventilation of the crankcase, the oil consumption was approximately the same as in the road tests.

In order to correct for expected engine change and the difficulty of resetting the engine to give exactly the same rate of consumption, all consumption data were determined and recorded as the ratio of consumption of the test oil to that of a reference oil. All tests consisted of three consecutive runs of two to three hours each without stopping the engine. The first and third were on the reference oil and the second was on the oil under test.

The oil-consumption data were plotted against time during each run. After the first few minutes of operation and if the operating conditions were held uniform, the plot was a straight line. The oil consumption, in pounds per hour, for each run was calculated from the slope of the plot. The deviation in the consumption of the reference oil in the first and third runs averaged less than one per cent. All tests in which this deviation was greater than three per cent were discarded. The consumption of the oil under test was reported as the ratio of its consumption to the average consumption of the reference oil for the first and third runs.

OILS USED

The volatility and viscosity characteristics of the test oils are shown in Table II. The first eleven oils were made from the same crude and have similar viscosity characteristics. In order to obtain a wide range in volatility, an S.A.E. No. 30 grade was used and all eleven oils were blended to a Saybolt Universal viscosity of 235 sec. at 130 F. This gave an average viscosity of 63 sec. at 210 F. The exact viscosities at 130 F. and 210 F. are given in Table II. The first eight oils are blends of bright stock, having a viscosity of 145 sec. at 210 F., with a series of paraffin pale oils, having viscosities of from 70 to 430 sec. at 100 F. The ninth oil is an experimental sharply fractionated distillate of the given viscosity.

Oils Nos. 10 and 11 are from the same crude source and have the same viscosity, but are blended with broad cut pale oils to give different over-all distillation curves than the first nine oils.

Oils Nos. 12 and 13 are from different crudes and have different viscosity characteristics. They were both blended from commercial S.A.E. Nos. 30 and 40 motor oils to have the same viscosity at 225 F. as oil No. 6-S, that is, 9.2 centistokes or approximately 55½ sec., Saybolt. Oil No. 12 is of Gulf coast crude origin, while No. 13 is an eastern paraffinic oil.

The distillation of the test oils under 2-mm. pressure was conducted in the Bureau of Mines type of equipment using a Cenco vacuum controller and a butyl-phthalate gage. The maximum pressure variation was a ± 0.2 mm. and the temperature was corrected for a variation of 0.1 mm. or more with a large scale Watson and Wirth chart.[1] The distillations of all oils are given in Table II. A comparison of the initial boiling points and the 2½ to 30 per cent distillation points will show the wide range of volatility covered by the oils tested.

TABLE II.—CHARACTERISTICS OF THE OILS USED.

	Oil No. 1	Oil No. 2	Oil No. 3	Oil No. 4	Oil No. 5	Oil No.6-S	Oil No. 7	Oil No. 8	Oil No. 9	Oil No. 10	Oil No. 11	Oil No. 12	Oil No. 13
Viscosity, Saybolt Universal, sec.:													
Neutral oil used at 100 F.	70	100	150	180	200	225	240	430	555	495	300	[a]	[a]
Blended oil at 130 F.	235	235.5	235	235	235	237	235	235	236	235	235	277	210
Blended oil at 210 F.	63.9	63.8	63.0	63.0	63.0	63.5	63.0	63.0	62.4	62.8	62.8	63.0	62.4
Percentage of neutral oil used in blend	37	43	44.5	55.7	58	62	60.7	86.1	100	92.3	69	......	.. ..
Flash point, deg. Fahr.	380	405	420	430	425	440	435	445	475	430	450	415	445
Fire point, deg. Fahr.	440	460	475	485	490	505	500	520	555	500	505	480	510
Distillation at 2 mm., deg. Fahr.:													
Initial boiling point	336	359	369	380	390	408	418	444	496	404	410	354	410
2½ per cent	354	375	392	405	411	430	434	475	511	431	426	385	429
5 per cent	370	389	410	422	426	446	447	498	522	451	440	410	443
10 per cent	388	414	430	438	444	462	464	522	536	469	458	442	459
20 per cent	414	434	454	465	468	483	487	552	554	496	482	477	487
30 per cent	428	451	480	485	486	500	505	577	568	522	499	500	505
40 per cent	502	484	506	504	506	520	523	599	579	543	517	519	525
50 per cent	570	545	540	528	530	539	542	620	589	562	536	535	543
60 per cent	608	592	580	562	557	563	564	640	600	580	558	552	564
70 per cent	662	642	630	604	592	598	594	662	608	600	588	570	594
80 per cent	700	690	684	664	646	658	640	685	620	628	632	592	662
90 per cent	[b]	[b]	[b]	[b]	718	[b]	708	[b]	636	672	706	622	[b]
$(T_{80} - T_5)$	330	301	274	242	220	212	193	187	98	177	192	182	219
Initial boiling point + 0.2 $(T_{80} - T_5)$	402	419	424	428	434	450	457	481	516	439	448	390	454

[a] Special

[b] Cracked.

As oil No. 6 was available in quantity and was being used on road test work, it was selected as the standard reference oil and will hereafter be designated as 6-S.

TEST RESULTS

The consumption data obtained by the Chevrolet engine on the above series of oils are given in Table III. The first column, oil number, refers to the same classification as given in Table II. The relative consumption of the oil under test is given as the percentage of the average consumption of oil No. 6-S obtained in runs Nos. 1 and 3 for each test. The percentage deviation of oil No. 6-S for the two runs of each test is given. The consumption of oil No. 6-S given in the line for that oil is the average of all tests.

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[1] K. M. Watson and Charles Wirth, III, "Routine High-Vacuum Distillation of Oils," *Industrial and Engineering Chemistry,* Analytical Edition, Vol. 7, No. 1, January 15, 1935, pp. 72-75.

The absolute consumption of oil No. 6-S for all runs is not directly comparable as occasional engine overhauls changed the basic consumption. In the 50 m.p.h. series all runs are directly comparable except for oils Nos. 8, 12 and 13. These latter were run some weeks after all other work was completed and after an engine overhaul. It is interesting to note the accuracy with which oil No. 6-S was checked between the two runs in each test. This indicates that the method for flushing the engine definitely overcomes any contamination through drainage between runs.

TABLE III.—CONSUMPTION TESTS WITH CHEVROLET ENGINE.

Oil	Relative Consumption, percentage of Oil No. 6-S	Consumption of Oil No. 6-S, lb. per hr.		Deviation Between 2 Runs on Oil No. 6-S, per cent
		Run No. 1	Run No. 3	
OPERATION EQUIVALENT TO 50 m.p.h.				
No. 1	149	0.1005	0.1011	0.6
No. 2	125	0.1006	0.1009	0.3
No. 3	121	0.0995	0.0990	0.5
No. 4	108	0.103	0.103	0
No. 5	103	0.103	0.103	0
No. 6-S	100	0.0923	0.0923	0
No. 7	97	0.103	0.104	1.0
No. 8	97	0.075	0.076	1.4
No. 8′	99	0.076	0.077	1.4
No. 9	91	0.0945	0.094	0.6
No. 10	116	0.104	0.1045	0.4
No. 10′	108	0.096	0.095	1.1
No. 11	106	0.1065	0.1076	1.1
No. 12	168	0.074	0.0735	0.7
No. 12′	164	0.075	0.074	1.4
No. 13	108	0.074	0.0722	2.5
OPERATION EQUIVALENT TO 60 m.p.h.				
No. 1	136	0.266	0.258	3.0
No. 2	119	0.251	0.255	1.6
No. 3	116	0.255	0.254	0.4
No. 4	104	0.255	0.252	1.2
No. 5	105	0.217	0.216	0.5
No. 6-S	100	0.226	0.225	0.5
No. 7	97	0.202	0.203	0.5
No. 9	94	0.199	0.197	1.0
No. 10	116	0.206	0.206	0
No. 10′	111	0.2005	0.199	0.7
No. 11	106	0.209	0.206	1.5
OPERATION EQUIVALENT TO 40 m.p.h.				
No. 1	140	0.0825	0.0832	0.8
No. 2	116	0.083	0.083	0
No. 3	107	0.084	0.083	1.2
No. 4	105	0.084	0.034	0.0
No. 5	104	0.0855	0.084	1.8
No. 6-S	100	0.0818	0.0817	0.1
No. 7	96	0.0825	0.0827	0.2
No. 9	94	0.082	0.0825	0.6
No. 10	112	0.069	0.0697	1.0
No. 11	107	0.083	0.083	0

The consumption reported may be considered as that resulting from the oil as charged, since there was practically no change in the viscosity or volatility characteristics during the test. A 250 end point straight-run aviation gasoline was used to eliminate serious crankcase dilution, and the test period was so short that there was no increase in viscosity due to oxidation or evaporation. The oil at the end of the test showed a viscosity change not greater than 2 sec. at 130 F.

Table IV gives the results for a similar series of tests made with an 8-cylinder in-line 120-hp. engine at 50 m.p.h. The actual consumption was higher in this series and the reproducibility not so good.

TABLE IV.—CONSUMPTION TESTS WITH 8-CYLINDER ENGINE.

Oil	Relative Consumption, percentage of Oil No. 6-S	Consumption of Oil No. 6-S, lb. per hr.		Deviation Between 2 Runs of Oil No. 6-S, per cent
		Run No. 1	Run No. 3	
No. 2	108	0.900	0.884	1.8
No. 3	115	0.765	[a]	...
No. 4	107	0.950	0.905	4.9
No. 5	102	0.993	0.949	4.6
No. 6	100	0.937	0.939	0.2
No. 7	99	0.918	0.882	4.1
No. 10	109	1.040	1.041	0.1
No. 11	106	0.990	0.974	1.6

[a] Not completed.

During September it became possible to make a few road runs with a Chevrolet test fleet of four cars. The fleet, freshly overhauled, was operated on oil No. 6-S for a 2500 mile break-in period. Two calibrating runs of 1000 miles were then made with oil No. 6-S to determine the difference in consumption between cars. A series of runs was then made comparing oil No. 6-S with the test oils Nos. 1 to 4, inclusive. In each run of 740 miles two cars were on oil No. 6-S and two on the unknown oil. The actual consumption was corrected for the car differences found in the calibrating run and the relative consumption of the unknown oil in terms of the percentage of consumption of oil No. 6-S was calculated. Table V gives the average results of two or more runs on each oil. The tests were over a long enough period to cause a material increase in the viscosity of the crankcase oil due to the evaporation of the more volatile oils. The average viscosity at 100 F. at the end of the runs is given in the same table. These viscosities should be compared with the original viscosity of all

TABLE V.—CHEVROLET FLEET ROAD TESTS.

Speed, 50 m.p.h. Water temperature, 180 F. Average oil sump temperature, 245 F.

OIL	RELATIVE CONSUMPTION, PERCENTAGE OF OIL NO. 6-S	AVERAGE VISCOSITY AT END OF TEST, SEC., SAYBOLT UNIVERSAL AT 100 F.
No. 1	146	900
No. 2	134	806
No. 3	143	737
No. 4	131	670
No. 6-S	100	631

oils of about 555 sec. at 100 F. While these tests were confined to fair days, the inevitable variation in temperature, winds, etc., made checks difficult.

DISCUSSION OF RESULTS

A study of the data in Table III indicates that there is a very definite increase in consumption with increase in volatility. In a crankcase oil it is probable that the first portion of the distillation range would have the greatest effect on consumption. The first nine oils, being of a uniform series, should show a reasonable correlation between their initial boiling points and consumption. This relationship is shown in Fig. 2 by the dots and heavy line.

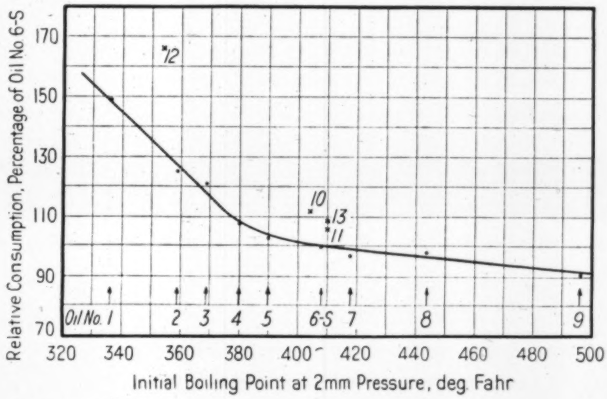

FIG. 2.—Relation of Consumption to Initial Boiling Point of Crankcase Oils.
Chevrolet engine at 50 m. p. h.

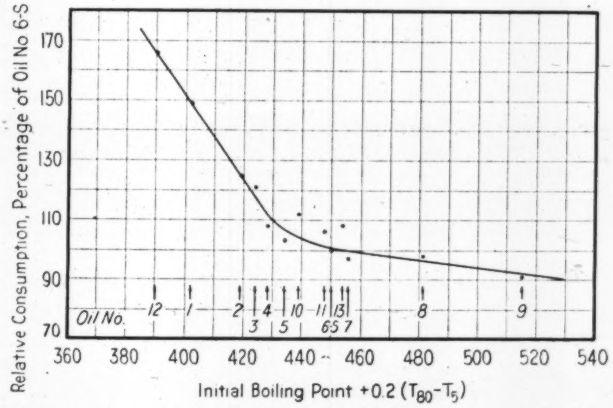

FIG. 3.—Relation of Consumption to Volatility Factor: Initial Boiling Point + 0.2 (T_{80}–T_5).
Chevrolet engine at 50 m. p. h.

The last four oils, not being of the same homologous series, show a definite non-conformity with this initial boiling point *versus* consumption curve as shown by the X's in Fig. 2. Similar graphs comparing the consumption with the $2\frac{1}{2}$, 5 and 10 per cent distillation points show lesser correlation, the higher the distillation point used.

The distillations of oils Nos. 10, 11 and 12 show a less over-all range as compared to those oils in the first series having similar initial boiling points. This characteristic is shown by the difference between the 80 per cent distillation point and the 5 per cent point in Table II. If this difference is significant, the initial boiling point modified by the over-all distillation range should show correlation with the consumption. Figure 3 gives the relationship between consumption and the initial boiling point plus one-fifth of the difference between the 80 and 5 per cent points: Initial boiling point $+0.2\ (T_{80} - T_5)$. This "volatility factor" shows good correlation for all oils except No. 13. There is no apparent variation in the distillation of the latter oil to explain this discrepancy, which is probably due to the error of test or some factor not yet recognized.

It is realized that with only one (No. 12) widely differing oil in the series the suggested volatility factor will probably require modification when additional data are available. The divergence of oil No. 12 may be partially due to viscosity differences, if the viscosity correlating temperature of 225 F. is not correct for the engine in question. A volatility factor using the slope for the first ten to thirty per cent evaporated rather than the over-all slope may give good correlation. It is possible that other factors such as surface tension, oiliness, molecular adsorption, etc. affect consumption and must be considered when comparing oils from different sources.

After the completion of the main series of tests at 50 m.p.h., runs were made at 40 and 60 m.p.h. on the Chevrolet engine. The consumption plotted against initial boiling point $+0.2\ (T_{80} - T_5)$ is shown in Figs. 4 and 5. In both cases the effect of volatility is not quite as great as at 50 m.p.h. This may be explained by the lower temperatures involved at the lower speed and the higher basic consumption at the high speed.

A similar correlation of the data obtained with the 8-cylinder engine and the Chevrolet cars is given in Fig. 6. The light line is the same as that given in Fig. 3. The dots and heavy line give the data for the 8-cylinder engine test. It is evident that volatility is of less importance in this case. The basic consumption was approximately ten times as great as in the previous tests. This confirms the opinion that the effect of volatility on consumption depends upon the actual basic rate of consumption.

The consumption obtained by the fleet road tests is shown by the X's. Although the correlation is not good, the general effect of volatility is of the same order as was obtained in the engine tests. The variation between oils is probably due in part to the variation in weather conditions

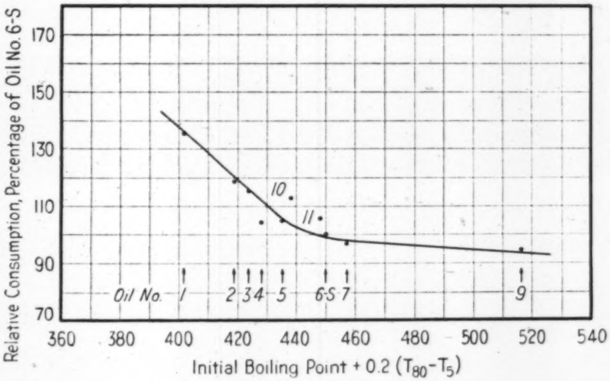

FIG. 4.—Relation of Consumption to Volatility Factor.
Chevrolet engine at 40 m.p.h.

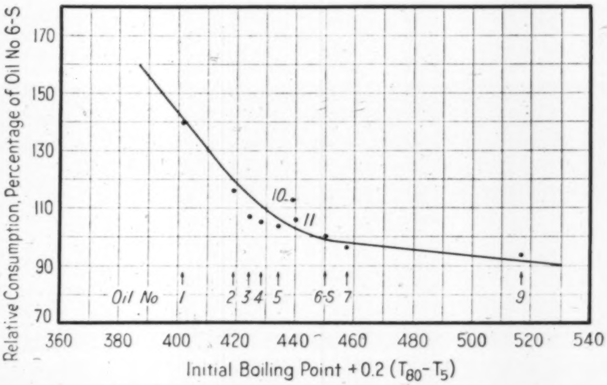

FIG. 5.—Relation of Consumption to Volatility Factor.
Chevrolet engine at 60 m.p.h.

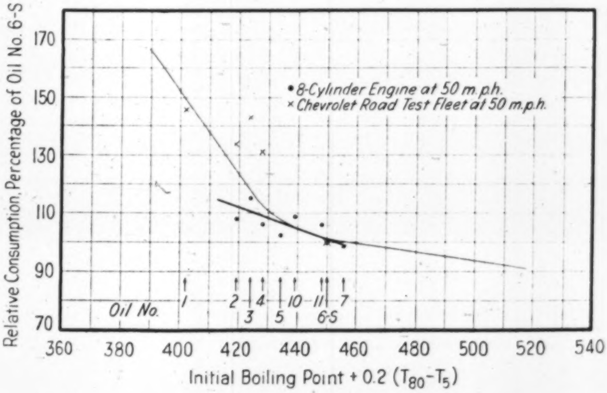

FIG. 6.—Relation of Consumption to Volatility Factor.

during the test. The change in viscosity during these runs also played an important role in the over-all consumption. This indicates the desirability of a short-time test as used in this work for the correlation of the various factors controlling consumption.

CONCLUSIONS

1. The effect of volatility as compared to viscosity on oil consumption appears greater than previously thought, particularly in cases of high-temperature operation where the basic consumption is low.
2. The effect of volatility on consumption is of minor importance when the consumption is very high because of engine design or high speed.
3. In a given uniform series of oil from the same source there is a simple relationship between front end volatility and consumption.
4. In an unrelated or non-uniform series of oils the volatility effect may be a function of the front end volatility modified by the over-all distillation characteristics.

It is felt that the problem is sufficiently important that some cooperative method should be established to continue this study. Not only should a more extensive series of oils be studied than was included in this report, but similar series should be prepared having the same volatility but different viscosities, so that the true relationship between volatility, viscosity and consumption can be determined.

DISCUSSION

MR. E. R. BARNARD[1] (*presented in written form*).—There is no question that consumption of motor oils in engines is to some extent governed by the volatility of its constituents. The question to be raised is simply, "To what extent does volatility influence consumption in motor oils?" The data presented by the authors would seem to indicate that volatility becomes an important factor only when an oil is made by blending a very light stock with a very heavy one to produce some intermediate viscosity. From Tables II and III of the paper it will be noted that only relatively small differences in consumption were recorded when the lighter blending stocks used had viscosities in the normal motor oil range, that is 150 Saybolt sec. and higher at 100 F. Oils Nos. 4 to 11, inclusive, all show consumptions within 8.0 per cent of the reference oil No. 6-S; or, in other words, the consumption figures of all of these oils lie within 5 per cent of the error limits set for the bracketing tests on oil No. 6-S. Within this range there is, in our opinion, more to be gained through improvement of viscosity temperature characteristics than through reduction in volatility.

That volatility of the lighter blending stocks can have a noticeable effect on consumption until such light ends have been duly eliminated from the main body of the oil is illustrated in the accompanying Fig. 1, which is a plot of oil consumption in terms of pounds per 100 miles at 60 m.p.h. against kinematic viscosity at 300 F. These data are from an 8-cylinder car used in actual road tests and the points are averages of two or more duplicate tests of 200 miles each. Oil A was made by blending with oil B about 8 per cent of a very light stock hardly heavier than the final fractions of U. S. motor fuel. In other words this oil is an aggravated form of the lighter oils used by Messrs. Davis and Best. Oil A shows an increase in consumption of 32 per cent over that of the base stock B, an increase that cannot be satisfactorily explained by lower viscosity alone. However, after 200 miles of operation the actual weight of oil A consumed was very little greater than that of oil B plus the 8 per cent of light blending material, and subsequent operation on the residue from oil A gave consumption figures closely approximating those of oil B. The same sort of performance is demonstrated by oils C and D on our chart. Here the light blending agent was used in somewhat smaller amounts, the base oil being somewhat heavier. The results are identical in form with those of oils

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A and B, the difference being only a matter of degree. We believe that if oils Nos. 1, 2, and 3, for example, in Table II of the paper had been run sufficiently long to drive off a fair proportion of the lighter ends, there would have been an improvement in consumption characteristics which would become decreasingly a function of volatility and increasingly a function of viscosity. Incidentally, whenever oil volatility is a predominating factor in consumption, a corresponding change in the crankcase oil due to loss of light constituents should be evident.

So far two oils in Table II have not been mentioned in this discussion, namely, Nos. 12 and 13. It will be noted that oil No. 12 is higher in viscosity at 130 F. than, for instance, is oil No. 7 although the viscosities at 210 F. are identical. The viscosity indices of oils Nos. 7 and 12 are 83 and 37, respectively, and their viscosities extrapolated to 300 F. are 39.7 and 38.8 Saybolt seconds or 4.1 and 3.7 centistokes. The difference in kinematic viscosity at 300 F. between these two oils is 0.4 centistokes. If this spread in viscosity be plotted on the accompanying Fig. 1 it will be seen that a considerable correction can be made for viscosity effect. For these oils in the engine used in the above tests the correction amounts to about 20 per cent, which places oil No. 12 on an equal footing with oil No. 1. If this curve had not changed slope so considerably before viscosities of the order of 4.0 centistokes had been reached the correction might have

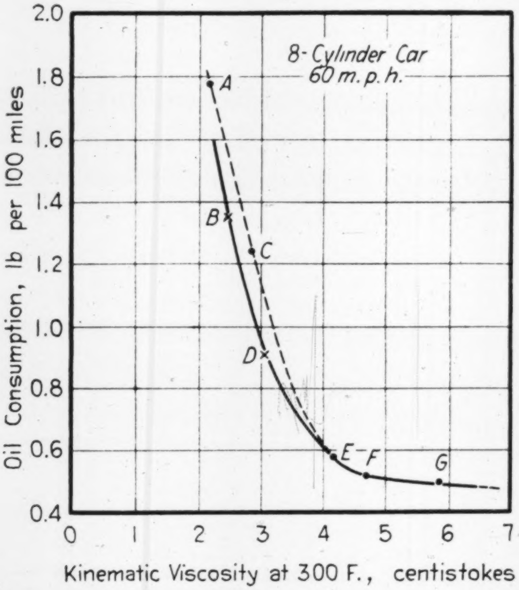

FIG. 1.—Relation Between Oil Consumption and Kinematic Viscosity.

been even greater. As such a curve for the engine used by Messrs. Davis and Best is not as yet available to us, we can only point out that in the case of oil No. 12, there might be an appreciable correction to be made in the data from this particular test.

From Table II it will be noted that oil No. 13 has excellent viscosity temperature characteristics. The viscosity index of this oil is about 102 and the kinematic viscosity at 300 F. is 4.2 centistokes. It is also one of the less volatile oils in the group, so that its consumption would be expected to be about the same or slightly less than that of oil No. 7. The data show slightly higher consumption although the difference is hardly out of the range of experimental error. In our opinion no average consumer of motor oils could definitely detect a difference in economy for oils Nos. 4 to 13 inclusive, with the exception of oil No. 12.

To sum up, it has certainly been indicated by the authors that volatility plays a part in the consumption characteristics of motor oils. In our interpretation this part is not as important as that of viscosity, particularly in oils made by reasonable methods of fractionation and in the viscosity range generally employed. They do point out, however, that a general swing to the use of light lubricants might make necessary the use of rather extreme blends as portrayed by oils Nos. 1, 2, and 3. In such an event volatility would evidently become of considerable importance. We believe their work to be a valuable contribution to the none too extensive knowledge of factors influencing oil consumption, and are happy to have had the opportunity to comment on their paper.

MR. J. P. STEWART.[1]—Viscosity and volatility are usually considered as the two oil characteristics which govern the rate at which an oil will be consumed in an engine, and numerous investigators have attempted to evaluate one or both of them. The usual type of engine oil consumption test is open to so many uncontrollable variables as to render difficult, results at all precise. Messrs. Davis and Best have employed a type of engine test which gives results of remarkable precision. By selecting oils of a given viscosity at a selected temperature they have attempted to eliminate viscosity effects, leaving the volatility characteristic as the controlling factor in the relative consumptions obtained, and from these data have set up an empirical formula for evaluating the effect of this characteristic. Even under the ideal conditions thus set up, Messrs. Davis and Best found one oil of the twelve tested that was not amenable to such regulation.

A certainty of being right 92 per cent of the time in the valuation of volatility would be a much happier condition than exists at present but can we expect that percentage?

In the particular set of data just presented all of the oils tested, with but two exceptions, are quite similar in make-up, being blends of a cylinder stock with various percentages of light stocks of differing viscosity and all

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[1] Head of Automotive Section, Research Division, General Laboratories, Socony-Vacuum Oil Co., Paulsboro, N. J.

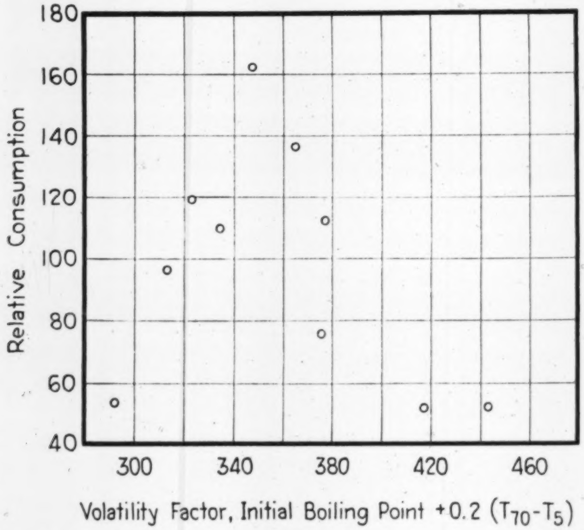

FIG. 2.—Relation of Consumption to Volatility Factor.

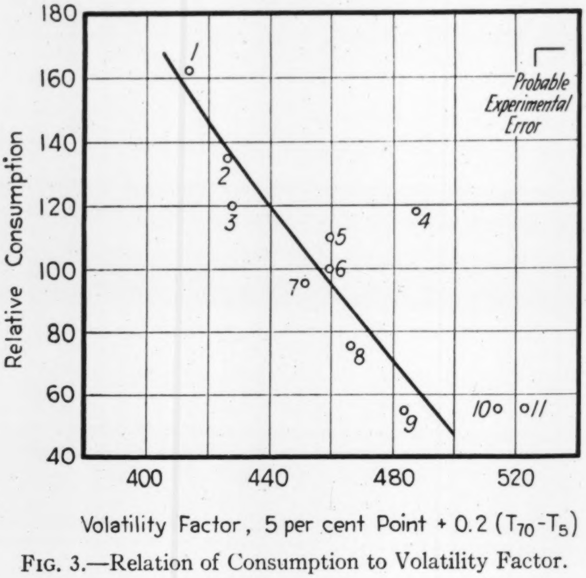

FIG. 3.—Relation of Consumption to Volatility Factor.

from the same crude oil. With such a group of oils one could expect a fair relation between consumption and several of the physical properties. It is therefore interesting to note that the one oil that does not conform to the empirical volatility formula is from an entirely different crude oil. What would have been the picture had the oils represented different crude oils and had been prepared by different refining methods.

The accompanying Figs. 2 and 3 present data obtained several years ago in a series of oil consumption tests employing the engine set-up referred to by the authors. The accompanying Table I gives the general make-up of the test oils, all of which were blended to the same viscosity at 210 F., with the exception of oils Nos. 9, 10, and 11 which were slightly lighter. In Fig. 2 the data are plotted in terms of Messrs. Davis and Best's volatility factor, except that the 70 per cent point was used instead of the 80 per cent point. Slight evidences of cracking were had at the latter point in the distillations of some of the oils, the distillation being made at 10 mm.

TABLE I.—CHARACTERISTICS OF TEST OILS.

OIL	VISCOSITY AT 210 F.	VISCOSITY INDEX	CRUDE OIL SOURCE	DESCRIPTION
No. 1	73.4	59	Coastal	Blend light and heavy stocks—conventionally refined
No. 2	74.4	93	Mid-Continent	Blend 155-sec. bright stock and 81-sec. neutral
No. 3	73.4	78	Mid-Continent	Blend 100-sec. bright stock (overhead) and 81-sec. neutral
No. 4	74.0	80	Mid-Continent	Blend of 130-sec. bright stock and 290-sec. neutral
No. 5	73.2	25	Coastal	Long overhead
No. 6	73.4	87	Mid-Continent	Blend 155-sec. bright stock and 150-sec. neutral
No. 7	73	87	Mid-Continent	Blend 120-sec. bright stock and 150-sec. neutral
No. 8	73.6	99	Pennsylvania	Blend of 120-sec. bright stock, 130-sec. neutral
No. 9	70	99	Mid-Continent	Reduced tar content by special laboratory still
No. 10	70	99	Mid-Continent	Long residuum Clearosol treated
No. 11	69	103	Pennsylvania	Lighter blend reduced

The shotgun pattern may or may not be due to the usual inaccuracy of the initial point in any distillation, yet check distillations gave initial points usually agreeing to within 20 F. The more logical reason is the fact that several of the test oils had very steep distillation curves up to the 2½ per cent to 5 per cent point and the small amount of light material represented by the low initial point could not possibly affect the consumption as a whole to any marked degree.

In Fig. 3 the data are again plotted in a similar manner except that the "Volatility Factor" was calculated on the basis of the 5 per cent point rather than the initial point. With this plotting a little more semblance of order is had, yet the departures of at least three of the test oils from the indicated curve is entirely outside the precision of the test, which is indicated in the upper right hand corner of Fig. 3.

It can be argued that the viscosity factor has not been eliminated from these curves for three of the oils differed slightly in viscosity from the others and the balance were blended to a uniform 210 F. viscosity whereas probable oil film temperatures were much above this figure. If a qualitative correction is made for viscosity at some higher temperature as indicated by the viscosity index of the several oils, the quasi-relationship indicated by the curve will be even less exact than that shown. Even a directional shift of oils Nos. 9, 10, and 11 in the plot of Fig. 3 to correct for the slightly lower viscosity of these three oils will not clarify the picture to any marked degree, the difference in viscosity being too small.

Probably no one will deny that viscosity and volatility are two characteristics of an oil that influence its consumption rate in an engine and the authors have probably come closer to evaluating the latter than has been done in the past. Yet all evidence points to at least one other characteristic at present not clearly defined. An evaluation of the oils covered by Figs. 2 and 3 as to their likely gumming effect discloses the fact that those most likely to cause gumming give the higher consumptions, those with the least tendency to gum give lower consumption rates. It is possible that the third oil characteristic affecting consumption may be found in this oil quality. In this connection we do not refer to actual sticking of the rings in the ring grooves, for the tests were of too short duration, but to an incipient formation that may slow down the ring action.

MR. A. L. CLAYDEN.[1]—Volatility unquestionably has a very distinct effect upon oil consumption. I think there is no doubt but that the viscosity index has a distinct effect. However, these are extremely insignificant items compared to differences in engines. It just so happens that within the last year my company also did some work with the Chevrolet engine. That engine, after taking it in its original conditions, after being limbered up with extraordinary care, entirely on the Indianapolis Speedway, on a particular S.A.E. No. 20-W oil, had a specific consumption of 600 miles per qt.

We then proceeded to do some simple mechanical work on that engine. We overcame oil leakage and changed the piston conditions by changing and very carefully fitting different types of rings. That immediately raised the oil mileage to 1360 miles per qt. That is an increase in mileage of 127 per cent, whereas, if you take the entire ranges of volatility explored by Messrs. Davis and Best you get a difference between the best and the poorest oil of 39 per cent on the dynamometer and 31 per cent on the road. I can see no reason why we should concern ourselves over minor differences in motor oils where there are still such enormous differences in engines.

I do not wish to give the impression that I think the authors' work is not worth while. I think it is very well worth while. I merely wish to emphasize that, due to the average new car being as it is, volatility is of very much smaller relative importance than would appear from tests made on very carefully prepared engines. In other words, if your average engine is running 600 to 700 miles per qt. and you fix it up so it is doing double that mileage, then you are exaggerating the effects of changing the oils, which is a very good thing to do if you want to study those changes, but I think it is extremely improbable that the average purchaser of motor oil would find volatility had anything like the same effect on his oil consumption that the authors obtained, or that would be obtained by similar carefully run laboratory tests. It appears to me we are still quite a few years off from the time when either volatility or viscosity index are of any serious importance as affecting oil consumption in the *average* car.

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[1] Research Engineer, Sun Oil Co., Philadelphia, Pa.

MR. R. E. WILKIN.[1]—I wish to emphasize that which Mr. Clayden has pointed out because I believe the charts in this paper may be misleading unless the entire paper is very carefully studied.

The data indicate variations in oil consumption due to differences in oil volatility of as much as 50 per cent. This may not have much significance in actual service when 25 to 50 per cent of the oil in the crankcase is consumed. The amount of oil used per hour in some of these tests was less than 0.1 lb., and if the test ran three hours the amount of oil consumed was less than 3 per cent of the total charge. Therefore, the first question that comes to my mind is: What would the oil consumption characteristics of these particular oils be had the tests extended for 10 or 20 hr. of running?

The other question I had in mind was whether the authors had run a vacuum distillation on the oil drained in order to determine what had been lost or what change had occurred in the volatility of the oil during its use.

MR. J. C. GENIESSE.[2]—It seems to me that in a comparison of the type made by Messrs Davis and Best, the ultimate relationship between volatility and consumption would depend on the amount of oil used during the experiment. For instance, if only a small percentage of the oil is consumed, we might get some relationship between the initial point or the 5 per cent point and the consumption of a motor. But if a large amount of oil is consumed, it is entirely possible that we would get a much better relationship between the oil consumed and some higher point on a distillation curve.

All of that brings out the point that the customer, it seems to me, is more interested in large amounts of oil consumed. He does not care whether he loses 2 per cent, 3 per cent, or 4 per cent. He is really interested in the possibility that he may lose 50 per cent of the oil, and I wonder whether this test procedure is really correct when we take that into consideration. I should like to have Mr. Davis' thoughts on that point.

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[1] Manager, Automotive Engineering Dept., and Assistant Manager, Technical Dept., Standard Oil Co. (Indiana), Chicago, Ill.

[2] Research Chemist, Research and Development Dept., The Atlantic Refining Co., Philadelphia, Pa.

MR. L. L. DAVIS[1] (*author's closure by letter*).—With reference to Mr. Barnard's discussion, I do not wish to give the impression that we question the importance of viscosity as the major factor determining oil consumption. We do, however, question his conclusions that the relationship between viscosity and consumption as plotted in his Fig. 1 isolates the true effect of viscosity. In such a series of oils, not only the viscosity but also the volatility of the oils varies over the entire range and therefore the consumption is the result of the combined factors. If the consumption of such a series of oils is plotted against volatility, an equally good relationship may be found—indicating that in actual practice both factors determine consumption. Assuming Mr. Barnard's viscosity correction for oil No. 12 to be correct, this oil still shows 40 per cent greater consumption than oil No. 6-S, which latter oil we would class as a good commercial product. As such highly volatile oils as No. 12 are actually on the market, we believe that volatility is of sufficient importance to be considered in evaluating commercial crankcase oils.

With reference to Mr. Clayden's remarks, we agree that far more can be done with engine design than with oil specifications to improve oil consumption, but until such changes are generally available, the car owner will certainly be interested in the oil that gives 25 to 40 per cent lower consumption.

In answer to Mr. Wilkin's comments, Table V and Fig. 6 of the paper give the results of long-time road tests. These show that the very volatile oils Nos. 1 to 4 showed 30 to 40 per cent higher consumption than the standard oil. In the short-time laboratory tests, both vacuum distillations and viscosity determinations on the used oil showed no appreciable change in the oil characteristics.

We agree with Mr. Geniesse that what interests the customer is the oil consumption over a long period of time. Practical tests must therefore include long-time road operations. Under such conditions, however, both viscosity and volatility change during the test, making it impossible to isolate the effects of either factor. Short-time laboratory procedure is therefore justified for the purpose of investigating the isolated factors effecting consumption.

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[1] Supervisor of Process Division, Continental Oil Co., Ponca City, Okla.

A FURNACE FOR THE MEASUREMENT OF COAL-ASH SOFTENING TEMPERATURE

By Elliott P. Barrett[1]

Synopsis

It is the purpose of this paper to describe a furnace designed for the specific purpose of determining coal ash softening temperatures and which is readily adaptable to measuring cone deformation temperatures in general up to 3000 F. (1650 C.). The furnace is of the horizontal muffle type and is so constructed as to permit the cones to be viewed against a background somewhat lower in temperature, so that they are readily visible at all temperatures between 1500 and 3000 F. (815 and 1650 C.).

The advantages of the horizontal muffle type of furnace over the conventional pot furnaces are emphasized. It is pointed out that the difference in design between the horizontal muffle furnace and the conventional pot furnaces does not affect the results obtained because the conditions in the former meet the requirements of the A.S.T.M. methods for determining ash softening temperatures.[2] The construction of the furnace is described in detail and is critically discussed. Comparative results obtained in this furnace with those obtained on the same ashes in pot furnaces are presented.

In conclusion, a critical discussion of the technique of ash softening measurements is presented.

Although softening temperatures of coal ash have been measured for a number of years in accordance with A.S.T.M. requirements,[2] no furnace especially adapted to this type of measurement has been described. The Denver Fire Clay and the No. 3 Melter's furnaces have been used probably because they were available, although they are not well adapted to the measurement of cone-deformation temperatures. The objections to the pot furnace, as typified by these two examples may be briefly summarized as follows: These furnaces are (1) very noisy, (2) very wasteful of gas, (3) so wasteful of heat as to make any ordinary laboratory excessively hot, (4) so designed as to make the observation of the cones well nigh impossible without disturbing conditions in the furnace, and (5) limited as to the number of runs which can be made in a day because their large heat capacity makes for slow cooling.

It is the purpose of this paper to describe a furnace designed especially for the purpose of measuring ash softening temperatures in accordance with A.S.T.M. methods and to compare results obtained with it and those obtained with the pot-type furnaces. This inevitably involves a general discussion of the sources of error in making such measurements. The type

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[1] Research Associate in Fuel Technology, Mineral Industries Experiment Station, The Pennsylvania State College, State College, Pa.

[2] Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 33), 1933 Book of A.S.T.M. Standards, Part II, p. 269.

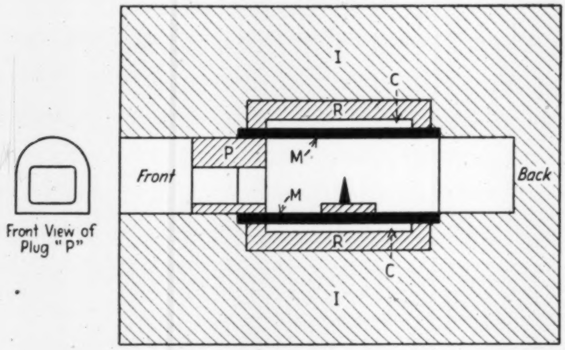

FIG. 1.—Vertical Section Through Center of Gas-Fired Muffle Furnace Designed for the Measurement of Coal-Ash Softening Temperatures.

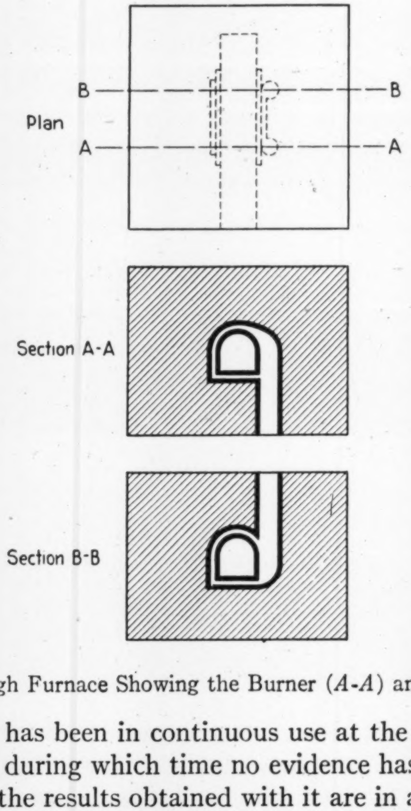

FIG. 2.—Sections Through Furnace Showing the Burner (A-A) and Exhaust Ports (B-B).

of furnace described has been in continuous use at the Experiment Station for nearly two years during which time no evidence has been obtained that would indicate that the results obtained with it are in any way less reliable than those obtained with pot furnaces.

Factors Affecting Furnace Design:

The factors to be considered in designing a furnace for ash-softening measurements fall into two groups: first, those involved in the requirements for the test; and, second, those involving facility of measurement, comfort of the observer, and economy of operation. If the design of the furnace is such as to meet the conditions imposed by the requirements of the test procedure, no departure, however radical, from the conventional pot-type furnace is of practical importance.

Description of the Furnace:

Figures 1 and 2 serve to illustrate the essential features of the furnace. It will be observed that many variations in detail are possible without in any way affecting the results obtained with the furnace.

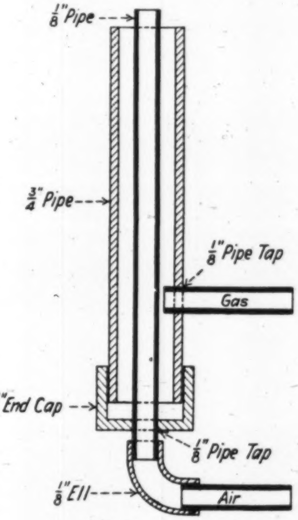

FIG. 3.—Vertical Section Through Center of Burner.

The figures show that the furnace is built around a "D" shape muffle, *M*, 2 in. inside width at the base and 2 in. inside height. The muffle is surrounded by a combustion chamber, *C*, $\frac{1}{4}$ in. in width and 4 in. long. The figures also show a refractory lining, *R*. This lining may be omitted if the furnace is not used above 2800 F. (1540 C.) but if it is desired to measure softening temperatures in excess of 2800 F. (1540 C.) the insulating brick must be protected or they will have a very short life. The insulation, *I*, is Babcock & Wilcox No. 80 insulating brick.

Sections *A-A* and *B-B*, Fig. 2, show the burner and exhaust ports, respectively. These enter and leave the combustion chamber tangentially so that in passing through it, the flame swirls smoothly around the muffle.

The ports are both of 1 in. internal diameter. An ordinary laboratory blast burner such as the Fisher No. 3-910 suffices to heat the furnace provided the gas jet in the bottom of the burner is removed, but a burner better suited to the low gas pressures generally available in laboratories is shown in Fig. 3. The only restriction on the burner is that it must provide a sufficient quantity of gas to heat the furnace at the specified rate with a sufficient excess of gas over air to maintain the specified reducing environment.

Critical Discussion of the Furnace:

It is apparent from Fig. 1 that the cones are heated by radiation from a uniformly heated muffle wall. That the cones are uniformly heated and, what is more important, that conditions in the muffle are essentially "black body" is easily shown. The furnace was designed with the intention of using an optical pyrometer sighted on the cones or cone pat as a temperature-measuring device. Repeated measurements of the melting points of gold and nickel using an optical pyrometer calibrated under black-body conditions have given results in excellent agreement with the accepted values for these constants. Such results show that the sight hole in the plug, *P*, and the rear extension of the muffle chamber into the insulation do not appreciably affect conditions at or near the longitudinal center of the muffle. Such being the case it is only necessary to show that the rate of heating can be kept within the specified limits while maintaining a reducing atmosphere within the muffle.

Because of the exceedingly small heat capacity of the furnace it is very easy to heat too rapidly even with a large excess of gas over air. Consequently, this problem reduces itself to a matter of skill and care on the part of the operator. The maintenance of reducing conditions within the muffle simply depends, then, on using a muffle sufficiently porous to permit the rapid diffusion of gas from the combustion chamber into its interior.

Two types of ceramic body have been found satisfactory from this point of view: a fire-clay body with a very course grog, and the Norton Company's R.A. 98 alundum. Using a muffle made of either of these materials, the diffusion of gases from the combustion chamber may easily be demonstrated by shutting off the air supply while the furnace is hot. Within a few seconds the muffle fills with flame due to the ignition of gas which has diffused through the muffle walls. When the air is again turned on the flame within the muffle disappears because the increased internal pressure prevents air from entering through the observation hole in the plug *P*. This result clearly indicates that, if the ratio of gas to air in the combustion chamber is properly controlled, the proper reducing conditions will be maintained in the muffle.

It is thus demonstrated that the matter of meeting specified conditions has been complied with.

In the matter of facility it is obvious that a furnace only 12 in. square and 9 in. high is a more convenient device than the large, heavy pot furnaces. Moreover, it is quiet enough to permit communication in an ordinary conversational tone. The outstanding feature, however, is the visibility of the cones. By means of the rear extension of the muffle chamber a thermal gradient is established which makes the cones readily visible at any temperature because they stand out as bright objects against a darker background. Nevertheless the radiation from the comparatively rough muffle walls supplies heat to the cones so rapidly that they do not cool appreciably either on the back or on the front where they lose heat by radiation through the observation hole.

TABLE I.—COMPARISON OF SOFTENING TEMPERATURE MEASUREMENTS MADE WITH DIFFERENT TYPES OF FURNACE.

Sample	Horizontal Muffle-Type Furnace		Pot Furnace		Differences				
					Horizontal Muffle-Type Furnace *versus* Pot Furnace			Horizontal Muffle-Type Furnace, Column 1 *versus* Column 2	Pot Furnace, Column 3 *versus* Column 4
	1	*2*	*3*	*4*	Column 1 *versus* Column 3	Column 2 *versus* Column 3	Column 1 *versus* Column 4		
No. 1	3000	....	2920	over 2800	+80	....	....	....	....
No. 2	2610	....	2620	2590	−10	....	+20	....	+30
No. 3	2310	....	2280	2260	+30	....	+50	....	+20
No. 4	2610	....	2520	2460	+90	....	+150	....	+60
No. 5	2710	....	2705	2670	+5	....	+40	....	+35
No. 6	2710	....	2670	2650	+40	....	+60	....	+20
No. 7	2325	....	2300	2210	+25	....	+115	....	+90
No. 8	2060	....	2040	2020	+20	....	+40	....	+20
No. 9	2710	....	2770	2680	−60	....	+30	....	+90
No. 10	2310	....	2370	2210	−60	....	+100	....	+160
No. 11	2155	....	2140	1960	+15	....	+195	....	+180
No. 12	2265	2310	2230	....	+35	+80	....	−45	....
No. 13	2250	2210	2255	....	−5	−45	....	+40	....
No. 14	2290	2260	2310	....	−20	−50	....	+30	....
No. 15	2430	2415	2415	....	+15	±0	....	+15	....
No. 16	2460	2470	2470	....	−10	±0	....	−10	....
No. 17	2550	2575	2590	....	−40	−15	....	−25	....
No. 18	2570	2585	2640	....	−70	−55	....	−15	....
No. 19	2680	2720	2700	....	−20	+20	....	−40	....
No. 20	2700	2700	2755	....	−55	−55	....	±0	....
No. 21	2770	2810	2800	....	−30	+10	....	−40	....
No. 22	2000	2015	2010	....	−10	+5	....	−15	....
No. 23	2175	2125	2110	....	+65	+15	....	+50	....
No. 24	2230	2160	2210	....	+20	−50	....	+70	....
No. 25	2425	2330	2390	....	+35	−60	....	+95	....
No. 26	2485	2385	2440	....	+45	−55	....	+100	....
No. 27	2450	2420	2510	....	−60	−90	....	+30	....
No. 28	2550	2540	2590	....	−40	−50	....	+10	....
No. 29	2695	2750	2770	....	−75	−20	....	−55	....
No. 30	2760	2810	2810	....	−50	±0	....	−50	....
No. 31	2590	2590	2525	....	+65	+65	....	±0	....
No. 32	2570	2615	2525	....	+45	+90	....	−45	....
No. 33	over 3000	....	over 3000	....	±0	....	....	....	....
No. 34	2680	....	2790	2680	−110	....	±0	....	+110
No. 35	2900	....	2950	....	−50	....	....	....	....
No. 36	2970	....	3000	....	−30	....	....	....	....
No. 37	2140	....	2145	....	−5	....	....	....	....
Average difference					±44			±37	+60
Within 45 F., per cent					56.0			71.5	50.0
Between 45 and 90 F., per cent					36.8			19.0	30.0
Between 90 and 135 F., per cent					4.2			9.5	0.0
Between 135 and 180 F., per cent					1.5			0.0	20.0
Greater than 180 F., per cent					1.5			0.0	0.0

Because of the quietness of operation, the relatively small amount of heat radiated to the laboratory, and the ease of observation, the comfort of the observer is greatly enhanced and the strain on him diminished. As a consequence, it has been found that three different observers at the Experiment Station consistently check one another to within 40 deg. Fahr. (22 deg. Cent.) or better.

As regards economy of operation, this type of furnace is infinitely superior to the pot furnaces. The gas consumption is greatly reduced and the replacement costs are much smaller. Moreover, the furnace cools rapidly so that many more runs per day are possible.

Comparison of Results:

Comparative results obtained in the muffle type of furnace with those obtained in pot furnaces for 36 samples of coal ash and one pyrometric cone powder are given in Table I. The results given in column 1 were obtained at the Experiment Station, those in column 2 at the laboratory of a coal company which had previously used a Denver Fire Clay furnace. Column 3, samples Nos. 1 to 30, inclusive, are the results of the Pittsburgh Experiment Station of the U. S. Bureau of Mines where the Denver Fire Clay furnace is used. Column 3, samples Nos. 31 to 36, inclusive, are results obtained in the laboratory of a coal company which uses the No. 3 Melter's furnace. The results in column 4, except sample No. 34, were obtained in the same laboratory as those of column 2 but a Denver Fire Clay furnace was used. Sample No. 34 was tested by a commercial testing laboratory using a No. 3 Melter's furnace.

Statistical Treatment of Results:

Although the data are too meager to justify the rigorous application of statistical theory it is, nevertheless, instructive to order the data in such a way as to facilitate interpretation. This has been done in Figs. 4 and 5.

In Fig. 4 softening temperatures obtained in horizontal muffle furnaces are plotted as ordinates against softening temperatures obtained in pot furnaces as abscissa. If conditions in the horizontal muffle furnace are substantially equivalent to those in the pot furnaces, the results plotted in this way should approximate a straight line of unit slope within A.S.T.M. tolerance. In Fig. 4 this theoretically perfect line has been drawn and two lighter lines 90 deg. Fahr. (50 deg. Cent.) above and below it are also shown. It will be seen that four points fall outside this band. That these four discrepancies are to be ascribed to faulty technique rather than to any peculiarities of the horizontal muffle furnace is demonstrable from Fig. 5 and Table I.

In Fig. 5 softening temperatures obtained in two different laboratories using horizontal muffle furnaces are plotted against each other as crosses.

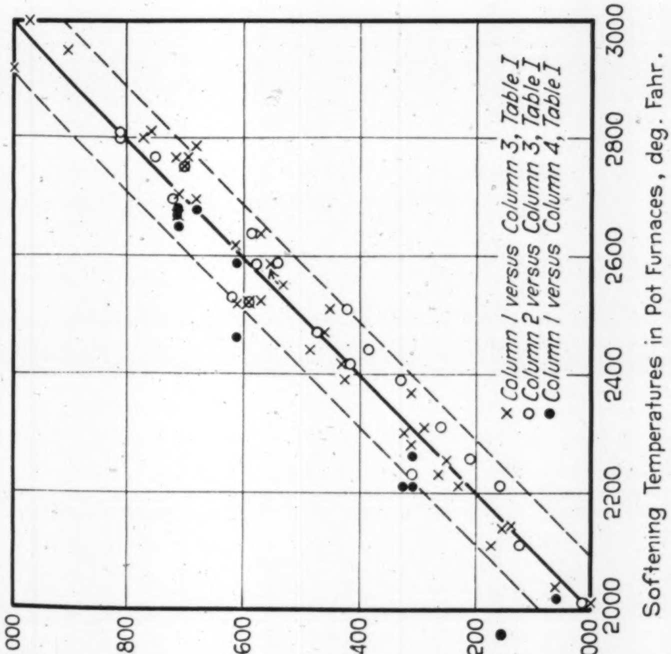

FIG. 4.—Showing Differences in Readings Made on the Horizontal Muffle Type and Pot Furnaces.

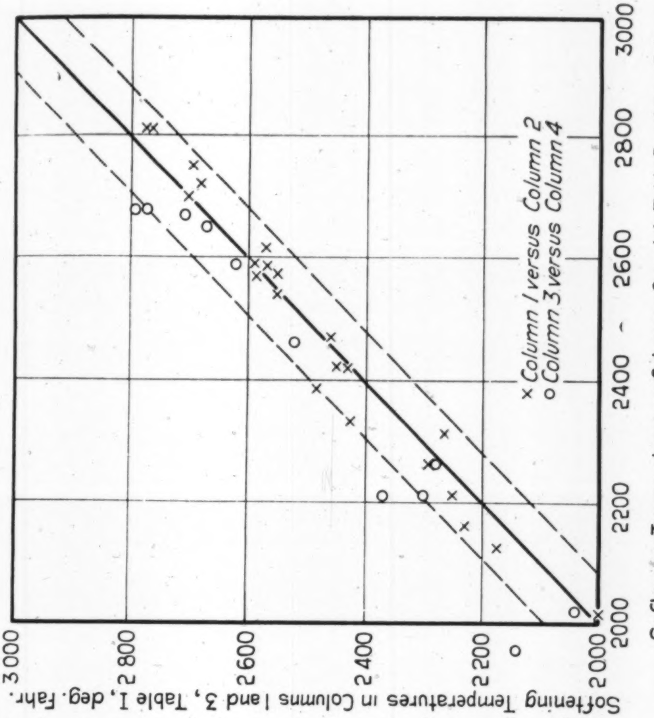

FIG. 5.—Showing Differences in Readings Made by Different Laboratories Using the Same Type of Furnace.

On the same plot softening temperatures obtained by two different laboratories using pot furnaces are plotted as open circles.

Table I gives the differences shown in Figs. 4 and 5. Both the figures and the table show that there is a symmetrical distribution of positive and negative differences at all temperatures. They also show that the laboratory using the pot furnace, listed in column 4, was consistently obtaining low results. The four points of Fig. 4 which fall outside the A.S.T.M. tolerance are all from this laboratory.

Critical Discussion of the Technique of Ash Softening Measurements:

It is of the greatest practical importance that different laboratories be able to obtain concordant results in this type of measurement. The author believes that with sufficient care the discrepancies between different laboratories can be reduced to 50 deg. Fahr. (28 deg. Cent.). As will be shown below, most of the large discrepancies which now occur can be assigned to lack of care and understanding on the part of those who make the measurements, but there is one respect in which the author believes the A.S.T.M. methods are deficient. Under the heading Preparation of Ash, the directions state ". . . . and completely convert to ash . . . at a temperature of 800 to 900 C."

It would be highly desirable to specify a definite time as well as a definite ashing temperature because there are a number of slow solid-phase reactions which can occur while the ashing process is going on. As examples of these the conversion of kaolinite to mullite and silica, the inversion of quartz to cristobalite, and the conversion of diaspore to corundum may be cited. In order that concordant results may be obtained, it is necessary that these reactions shall have proceeded to the same extent when the coal has been ashed in different laboratories. This condition is impossible of attainment unless a definite time limit is imposed. Our knowledge of solid-phase reactions is still limited, but there is available sufficient information to indicate that many solid-phase reactions proceed at surprisingly low temperatures. Consequently, some attention must be paid to the possibility of discrepancies arising from this source.

In the category of discrepancies due to lack of care and understanding on the part of those making the measurements, occur the following: Failure to grind the ash finely enough and to pack the cones tightly into the mold, failure to utilize temperature measuring equipment correctly, and failure to maintain a reducing atmosphere around the cones. The author recalls a number of instances in which ash has been sent to him by other laboratories, with a request for a check measurement, which was so coarse that it gritted between the spatula and the glass plate in mixing the ash with water preparatory to molding cones. In another instance in checking the sources of a discrepancy the author found that a laboratory using the

Denver Fire Clay furnace with a thermocouple had placed the couple so far away from the cones that there was only the faintest chance of obtaining correct temperature readings.

It is in this matter of correct temperature readings that the horizontal muffle-type furnace demonstrates one of its marked superiorities over the pot furnaces. By sighting an optical pyrometer on a cone or on the cone pat at the base of the cone, the observer is assured that he is reading the true temperature of the cone as nearly as human limitations permit. When using an optical pyrometer with the pot-type furnaces, the observer is merely looking into a glare of light which he hopes bears some relationship to the temperature of the cones.

The author believes that the use of thermocouples should be discouraged because it is difficult so to place them in the furnace that they will give the true temperature of the cones without also obstructing vision.

There are certain minor points which seem worth mentioning. The first of these is the matter of cone size. The author has used cones of the standard proportions but has varied the height all the way from the standard $\frac{3}{4}$ in. down to one half the standard height without obtaining any appreciable difference in results. The object of using smaller cones was to be able to get more cones into the furnace. The first eleven samples of Table I were run simultaneously using half-height cones. However, experience indicates that six cones are about as many as an observer should try to watch and that a height between three fourths and seven eighths of the standard is easier to see clearly than the half-height cones. With a furnace of the dimensions given in this paper there is less crowding if the somewhat smaller cones are used when six are mounted on the same pat.

Another point of interest is that of the pat material. The A.S.T.M. methods call for a pat of calcined alumina and kaolin. It seems to the author that it is a waste of time to make up a special ceramic body of a special shape each time a measurement is made. The practice at the Experiment Station has been to cut a rectangular block of Babcock & Wilcox No. 80 insulating brick $\frac{1}{4}$ in. thick as a base. This material can be cut very easily with a hack saw. A layer of wet alundum cement about $\frac{1}{8}$ in. thick is spread upon the base and the cones pushed down into it. It requires less than a minute to prepare such a pat and the alundum cement will not react with the slag so rapidly as will a material containing a relatively large amount of silica as does kaolin.

SUMMARY

1. The pot-type furnace is a crude and unsatisfactory device for the measurement of coal-ash softening temperatures.

2. The horizontal muffle-type furnace can be so designed as to be an efficient, facile, and accurate apparatus for this purpose.

3. The optical pyrometer is to be preferred to the thermocouple as a temperature measuring device in ash softening measurements.

4. Of the many sources of error the failure to maintain a reducing atmosphere is the most serious and is of frequent occurrence.

5. The exact size of cone used appears to be of little importance.

6. As a material for mounting the cones, a high-alumina refractory is to be preferred to one containing considerable amounts of silica.

7. The use of a dextrin binder should be discontinued. When one has learned to make cones properly it is just as easy to make them with water. The dextrin is merely a time-wasting complication.

Acknowledgment.—The author desires to thank the Central Pennsylvania Coal Producers' Association for permission to publish this material.

DISCUSSION

MR. W. A. SELVIG[1] (*presented in written form*[2]).—Pot-type furnaces such as the coal-ash fusion furnace of the Denver Fire Clay Co. and the No. 3 Melters furnace of the American Gas Furnace Co. have been used successfully for coal-ash fusion determinations at the Pittsburgh Experiment Station of the U. S. Bureau of Mines for 18 yr. During this period these furnaces have been in almost daily operation, and thousands of coal-ash fusion tests have been made. It is true that these furnaces occupy considerable space, are noisy in operation, use relatively large amounts of gas, and cool slowly after each run owing to their large heat capacity. I believe, however, that Mr. Barrett is too severe in his criticisms of these pot-type furnaces.

The Denver Fire Clay furnace, which was designed for this test, is well insulated and does not radiate as much heat to the laboratory as does the No. 3 Melters furnace. Two Denver Fire Clay furnaces are installed at the Pittsburgh Experiment Station in a laboratory of moderate size. Sheet-iron canopies connected to an exhaust system are placed over the furnaces to conduct the hot gases out of the room. Only during the hot days of summer is there any discomfort to the operator due to heat radiated from the furnaces.

As correct temperature measurements with an optical pyrometer can be readily made in a furnace that closely approximates black-body conditions, it is surprising that the author should claim that the pot-type furnaces are not well adapted to correct temperature measurements of the ash cones. The fact that the ash cones are more visible in his furnace means that this type of furnace departs further from black-body conditions than does the pot-type furnace. The data given in the paper indicate, however, that this greater departure from black-body conditions does not have an appreciable effect on the temperature measurements of the ash cones. At the lower temperatures the individual ash cones in the pot-type furnace are visible when viewed through an optical pyrometer, and temperature measurements can be made by sighting on the ash cones. At the higher temperature the conditions in the pot-type furnace so nearly approach those of a black body that the ash cones are not visible through an optical pyrometer; then correct temperature measurements can be made by sighting the pyrometer in the immediate vicinity of the ash cones.

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[1] Chemist, U. S. Bureau of Mines, Pittsburgh, Pa.

[2] Published by permission of the Director of the U. S. Bureau of Mines.

Both thermocouples and optical pyrometers have been used by the Bureau of Mines for measuring the temperatures of the ash cones. No difficulty has been experienced in installing thermocouples in pot-type furnaces to obtain correct temperature measurements. Thermocouples should not be used if the furnaces are operated at temperatures higher than 2730 F. (1500 C.). As the Bureau tests many coal ashes softening above 2730 F. (1500 C.), it has standardized on the use of optical pyrometers. From the standpoint of ease of manipulation, we prefer to make temperature measurements by means of an optical pyrometer.

The device for momentarily cooling the ash cones in the pot-type furnace by means of a jet of compressed air to make them more readily visible when the deformation points are observed does not have any deleterious effect on the results of the test as far as can be determined. The ash cones, of course, are never cooled when temperature readings are made, but only when a critical examination is made to determine the state of fusion of the ash cones. Our experience is that the skilled operator seldom needs to use this cooling device, as his eye becomes trained to detect the deformation points of the ash cones even at the higher temperatures when they are only faintly visible.

In regard to possible reactions between the ash cones and the kaolin in the material in which the cones are mounted, we have not found any indications that such reactions proceed far enough to be detected in the fusibility determination. Tests have been made with coal ashes of high iron content and low fusibility by mounting the cones in the regular manner and also by simply placing them on platinum, and no differences in softening temperatures could be detected. The method of mounting the ash cones as described by the author should be satisfactory, but it does not appear to be any improvement over the method as given in the standard method either in regard to speed of mounting or in regard to accuracy of the determination.

The author speculates as to solid-phase reactions that may occur during the ashing of the coal but offers no data to show that such reactions have any influence on the determination of the softening point of coal ashes. Unless it can be shown experimentally that such reactions have a significant bearing on the fusibility determination it does not seem advisable to require that coals must be ashed for the same length of time.

The horizontal-type muffle furnace which the author describes has a number of desirable features and I do not intend to imply that it is not entirely satisfactory for coal-ash fusion tests. According to the manufacturers of this furnace it can be operated satisfactorily on cylinder gases on account of its low gas consumption. This should be an important item in laboratories where manufactured or natural gas is not available or, if available, the price is too high for economical operation of large pot-type furnaces.

MR. H. C. PORTER[1] (*presented in written form*).—The furnace described in the paper appears to offer a number of important advantages over the present standard pot type, and it has so much promise that I hope further extended trials may be made of it by a number of laboratories. The author's claims as to economy of gas and of replacements, increased capacity, less noise and heat and space requirement, all seem to be justified; but I cannot quite agree with him that the present standard pot-type furnaces are "so designed to make the observation of the cones well-nigh impossible without disturbing conditions in the furnace." Through a long experience in using the present standard furnace, I have had almost no difficulty on this account, with tests running at times up to 2900 F. (1595 C.).

It must be recognized that this standard method of test is an empirical one, with a definitely fixed set of conditions which are known to have an important effect on the results. It does not purport to set up conditions duplicating those of industrial furnaces nor to obtain results as to ash softening points duplicating what would obtain in practice on the same coals or refractories. It is therefore entirely possible for a new test equipment to be substituted as standard, provided it gives a reasonable approximation to the conditions prevailing in industrial furnaces (particularly as to reducing atmosphere), and fulfills requirements for the necessary accurate control of conditions, and a minimum disturbing effect of the slight variations in operating method and in conditions that must be expected in the hands of different operators and laboratories. It is also possible that a new type of furnace such as this might be made an alternate method provided it can be shown to check well against the present standard furnace in different laboratories.

It would have been of interest and value if the author had shown results of tests to indicate the magnitude of effect of variation in conditions, such as atmosphere or flame type, position of the cone-block in the muffle, fineness of pulverization of the ash, use or non-use of preoxidation of the powdered ash, etc. A more convincing demonstration of a reducing atmosphere within the muffle during a run would add weight to the argument. As a practical matter of upkeep, it would be of interest to know the replacements necessary of the muffle when a material sufficiently porous for gas penetration is used.

The results in Table I show, in 37 tests checking against the standard pot furnace (chiefly at the Bureau of Mines), an average deviation of only 44 deg. Fahr., but 17 were more than 50 and there were individual deviations of 195, 150, 110, 90, 75 and 70; also, in column 2 the checks in another laboratory with the author's furnace show good average agreement (37 deg. Fahr. in 21 tests), but on 8 low-fusing ashes (under 2400 F.) presumably high in iron and affected by reducing conditions, there was an average deviation of 56 deg. Fahr. Further check by several laboratories is needed to clear up this uncertainty.

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[1] Chemical Engineer and Chemist, Philadelphia, Pa.

MR. O. O. MALLEIS[1] (*presented in written form*).—The experience of the writer in determining ash softening temperature has been entirely with the present pot-type furnace. He did, however, observe a demonstration run by Mr. Barrett of his new furnace at State College in the fall of 1934. The most outstanding feature of Mr. Barrett's furnace was the better visibility of the cones. Other apparent advantages over the pot-type furnace were: (1) more rapid cooling and therefore more runs per day; (2) much smaller gas consumption; (3) quieter operation and (4) less heat radiated.

The values obtained by the two laboratories using the Barrett furnace (columns 1 and 2 of Table I) are of the same order of magnitude as those obtained by the present pot-type furnace (column 3). Also the plus and minus differences between columns 1 and 2; 1 and 3; and 2 and 3 are fairly equally distributed. It is, therefore, surprising that all the values in column 4 should be higher than those of either columns 1 or 3, with some of the differences abnormally large for check tests on the same ash sample. Possibly there was some constant error in obtaining the values in column 4 that caused all the differences to be in the same direction.

Since the results obtained by the Barrett furnace are apparently of the same order of magnitude as those obtained by the present pot-type furnace, and since the Barrett furnace apparently has many advantages over the pot-type furnace, it would seem desirable that Committee D-5 on Coal and Coke make a complete investigation of this new furnace. If found satisfactory, this furnace should then be recognized as suitable for the determination of coal-ash softening temperatures.

MR. J. B. ROMER.[2]—The furnace described by the author has the advantage of having been developed for a specific purpose, whereas the commonly used Melter's and Denver Fire Clay furnaces were designed for other purposes.

The use of an extension into the rear of the furnace raises the question as to whether true black-body conditions are maintained; however, the average agreement between different tests, as shown in Table I of the paper, indicates that the individual variations as shown are due either to the personal equation or the variation in the sample rather than to any inherent error in the new type of furnace.

The author has taken advantage of newly developed insulating brick in constructing his furnace, and thereby obtains greater gas economy and at the same time is enabled to operate his furnace with an ordinary blast burner. The low heat capacity of the insulating brick which he used, permits rapid cooling of the furnace, hence many more runs per day per furnace. A Melter's furnace has a 2-in. wall and at 2800 F. (1540 C.) such a wall, when made of firebrick, has a heat content of approximately 8600 B.t.u. per sq. ft., as compared with a value of approximately 2000 B.t.u. per sq. ft. for a similar wall made of the same insulating brick as used by Mr. Barrett in constructing his furnace. I consider that the proper application of that same insulating brick to a Melter's furnace or to a Denver Fire Clay furnace would greatly improve the economy of these furnaces, and also permit more runs per day.

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[1] Manager, Inspection Division, Appalachian Coals, Inc., Cincinnati, Ohio.
[2] Chief Chemist, The Babcock & Wilcox Co., Barberton, Ohio.

MR. J. F. KOHOUT.[1]—We have been using one of these Barrett furnaces for approximately six months, and comparing the results obtained in that furnace with electric furnaces of our own design which we also installed in Chicago and in our Charleston laboratory, where they also have a Denver furnace, the results on the four furnaces checked well within the tolerances of the Society's standard method.

In addition, a good many samples have either been sent to the Pittsburgh Station of the Bureau of Mines or sent to us by them, and those samples have also fallen right in line. Our experience has been very satisfactory with the furnace recently. Before that we were trying to operate with a Maxon premix burner and were not able to get much over 2700 F. (1480 C.). With the Barrett blower we are able to get the high temperatures. Our one trouble is that the furnace is quite noisy and the blower whines at high speed.

MR. ELLIOTT P. BARRETT[2] (*author's closure by letter*).—The author wishes to express his appreciation for the friendly and instructive discussion which has been given, but feels that he must take exception in part to two points in Mr. Selvig's discussion.

It is not necessarily true that a pot-type furnace, such as the Denver Fire Clay, will always approximate closely a black body. If such a furnace is held at a particular temperature for sufficient time for all of its contents to reach the temperature of its walls, then it will very closely approximate a black body and it will make no difference at what point within the furnace the temperature is measured. However, when the furnace temperature is changing, as is the case when making an ash-softening measurement, thermal gradients can and do exist within the furnace and the point at which the temperature is taken becomes of importance.

This is admirably illustrated by a point which Mr. Malleis has raised where he expresses surprise that all the values in column 4 of Table I are higher than those of column 3, Table I. This circumstance arose from the fact that the operator who obtained the results of column 4 placed the cones at the very front of the inner crucible while the thermocouple junction was well back in the crucible. Since, under these conditions, he always obtained results higher than those of the Pittsburgh Experiment Station of the Bureau of Mines, it must be supposed that there existed a thermal gradient from the couple junction to the cones.

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[1] Director of Laboratories, Commercial Testing and Engineering Co., Chicago, Ill.

[2] Research Associate in Fuel Technology, Mineral Industries Experiment Station, The Pennsylvania State College, State College, Pa.

With reference to the matter of ashing for a definite length of time, the writer feels that there can be little doubt that a careful investigation would demonstrate the desireability of such a practice. However, such an investigation would be expensive and time consuming out of proportion to its value and it might be better simply to adopt a standard time without investigation.

In the matter of a mounting material, the author did not mean to imply that the kaolin-alumina base would seriously affect the results, but merely that it involved an unnecessary amount of trouble.

I am sorry that I was not in position to present data supporting the contention that the different crystal modifications, in which various oxides may appear in the ash, are significant. I have data of that character which I have not as yet been given permission to publish. Mr. S. S. Cole now holds a patent for raising the softening temperature of coal ash by the addition of Al_2O_3 to the coal. He found that this treatment frequently raised the softening temperature, provided that the coal was ashed at 750 to 800 C., but if the coal were treated in this way and then coked at 1000 C. the softening temperature was not raised at all.

THE DISTRIBUTION AND VARIATIONS OF CERTAIN STRENGTH AND ELASTIC PROPERTIES OF CLEAR SOUTHERN YELLOW PINE WOOD

By A. W. Brust[1] and E. E. Berkley[1]

Introduction

The classification of structural timber into grades makes its use in construction safer, more effective and more economical. Improvements in methods of selection and grading are desirable to increase the economic use. Accurate strength classification of any one species requires a thorough knowledge not only of the average strength characteristics but also of the extent and distribution of strength variations in different trees and in different parts of the same tree. Most of the mechanical properties of clear wood are known to be closely related to the density. A number of these relations, determined from the average densities and strength properties of each of many species of American woods, were put into algebraic form and the average deviation of any one species from the mean of all the species was determined by Newlin and Wilson **(4)**.[2] Data in this form are indispensable for determining the usefulness of a particular species for a specific use or for selecting the best species for a given set of conditions. Knowledge of the variability within a species and its causes is quite as important as the average strength properties of the species, since such variation is usually greater than the difference in the average values of the various species which may be considered for a specific use.

An attempt is made here to evaluate and describe some of the variations in structure which affect the strength-density relations in the species of pine represented. By making allowance for differences in cell structure in addition to the density or percentage of summerwood, it is possible to judge the mechanical properties of a stick of timber more accurately. It is not desired that the usefulness of the density or percentage of summerwood for selecting timbers be reduced but rather that it be enhanced by a better knowledge of their limiting factors.

The principal factors generally recognized as affecting strength of clear, straight-grained wood are moisture and density. The density and thus the strength of clear wood has been judged almost entirely by the summerwood content with little emphasis on the structural quality of the wood. Study of the variation within the species shows that the quality of the clear wood varies greatly in the same tree and that such variation is not wholly determined by the density and even less accurately by the summerwood content.

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[1] Assistant Professor of Civil Engineering, and American Creosoting Fellow in Botany and Civil Engineering, respectively, Washington University, St. Louis, Mo.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 673.

Berkley(1), by coordinating a study of the minute structure of southern yellow pine wood with its compressive strength, has shown that variations in the strength of clear wood not explained by variations in moisture and density are caused, for the most part, by differences in the structure of the wood and that such differences in structure are related to the particular species of pine, position in the tree, age of the tree, and general growth conditions. The present study, based largely on the same test material reported on by Berkley, deals with variations in bending as well as compressive strength, proportional limit and stiffness in compression and bending, and will attempt to point out some of the effects of local variations on the behavior of large timbers under stress.

TABLE I.—FIELD NOTES FOR THE SEVEN TREES STUDIED.

	Tree No. 1 Loblolly Pine	Tree No. 2 Loblolly Pine	Tree No. 5 Loblolly Pine	Tree No. 7 Loblolly Pine	Tree No. 3 Shortleaf Pine	Tree No. 4 Longleaf Pine	Tree No. 6 Longleaf Pine
Where grown	Near Tylertown, Miss.	Near Tylertown, Miss.	About 8 miles west of Sandy Hook, Miss., near Louisiana border	In Tylertown, Miss.	Near Tylertown, Miss.	Near Tylertown, Miss.	Near Tylertown, Miss.
Approximate land elevation, ft.	225	250	225	...	265	300	...
Soil conditions	Moderately dry clay loam	Wet clay loam	Moist sandy clay	Sandy clay top soil, red clay sub-soil	Dry sandy clay	Dry sandy clay	Sandy clay top soil, red clay sub-soil
Exposure	West slope	Flat	Flat	Gentle south slope	Steep northwest slope	Steep southwest slope	Gentle north slope
Stand of timber	Dense second growth in old field	Moderately dense original swamp growth	Dense original hard wood with scattered pine trees	Open second growth hard and soft wood	Moderately dense original growth recently cut over leaving open stand	Dense original growth recently cut over	Open second growth recently cut over
Height of tree, ft	93	85	135	98	75	75	82
Diameter under bark at butt, in	18 (7 in. from ground)	18½ (30 in. from ground)	43	19½	17¾ (10 in. from ground)	16½	17½
Approximate age, yr	50	50	160	60	100	160	160
Rate of growth	Rapid	Rapid	Medium to rapid except for very slow growing outer 4 or 5 in.	Rapid	Moderately slow	Very slow	Very slow
Date of cutting	Oct. 4, 1930	Oct. 4, 1930	Oct. 2, 1931	Feb. 1, 1933	Oct. 4, 1930	Oct. 4, 1930	Dec. 6, 1932

TEST MATERIALS

The test material was taken from seven pine trees, all from Walthall County, Mississippi. Four of these trees were loblolly pine, *Pinus taeda*, two were longleaf pine, *Pinus palustris*, and one was shortleaf pine, *Pinus echinata*. Field notes taken by a representative of the American Creosoting Co. when the trees were cut are shown in Table I.

Test material from trees Nos. 1, 2, 3, and 4 included the first 32 ft. above the stump. Material from trees Nos. 5, 6, and 7 consisted of three

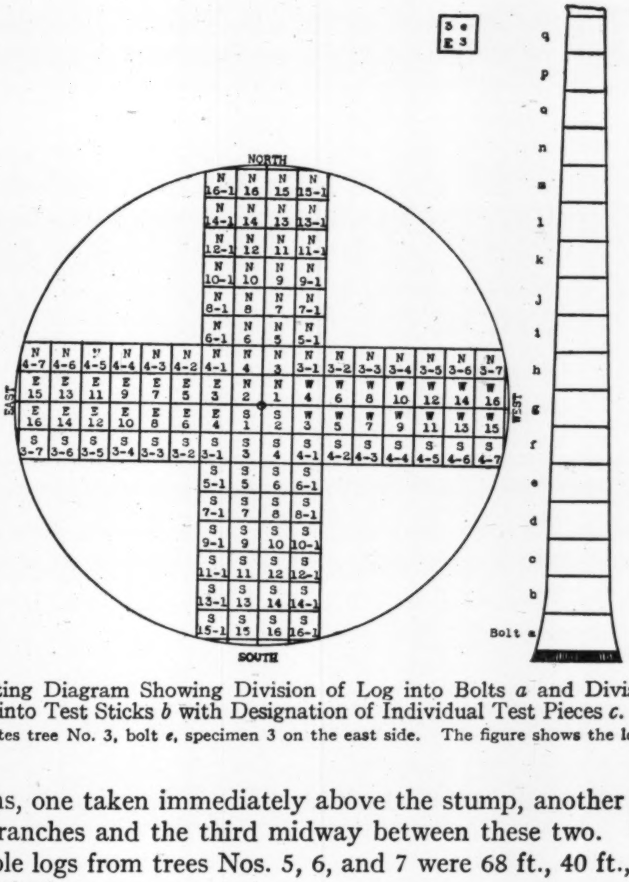
3e
E3
NORTH
N 16-1 | N 16 | N 15 | N 15-1
N 14-1 | N 14 | N 13 | N 13-1
N 12-1 | N 12 | N 11 | N 11-1
N 10-1 | N 10 | N 9 | N 9-1
N 8-1 | N 8 | N 7 | N 7-1
N 6-1 | N 6 | N 5 | N 5-1
N 4-7 | N 4-6 | N 4-5 | N 4-4 | N 4-3 | N 4-2 | N 4-1 | N 4 | N 3 | N 3-1 | N 3-2 | N 3-3 | N 3-4 | N 3-5 | N 3-6 | N 3-7
E 15 | E 13 | E 11 | E 9 | E 7 | E 5 | E 3 | N 2 | N 1 | W 4 | W 6 | W 8 | W 10 | W 12 | W 14 | W 16
E 16 | E 14 | E 12 | E 10 | E 8 | E 6 | E 4 | S 1 | S 2 | W 3 | W 5 | W 7 | W 9 | W 11 | W 13 | W 15
S 3-7 | S 3-6 | S 3-5 | S 3-4 | S 3-3 | S 3-2 | S 3-1 | S 3 | S 4 | S 4-1 | S 4-2 | S 4-3 | S 4-4 | S 4-5 | S 4-6 | S 4-7
S 5-1 | S 5 | S 6 | S 6-1
S 7-1 | S 7 | S 8 | S 8-1
S 9-1 | S 9 | S 10 | S 10-1
S 11-1 | S 11 | S 12 | S 12-1
S 13-1 | S 13 | S 14 | S 14-1
S 15-1 | S 15 | S 16 | S 16-1
EAST
WEST
SOUTH
q
p
o
n
m
l
k
j
i
h
g
f
e
d
c
b
Bolt a

FIG. 1.—Cutting Diagram Showing Division of Log into Bolts *a* and Division of Bolts into Test Sticks *b* with Designation of Individual Test Pieces *c*.

3eE3 indicates tree No. 3, bolt *e*, specimen 3 on the east side. The figure shows the lower end of the bolt or log.

8-ft. sections, one taken immediately above the stump, another just below the main branches and the third midway between these two. The entire merchantable logs from trees Nos. 5, 6, and 7 were 68 ft., 40 ft., and 56 ft. long, respectively.

The test logs were shipped with the bark intact and the ends painted immediately after cutting. Upon receipt in St. Louis, the logs were marked and cut into test specimens according to the schedule indicated in Fig. 1. Specimens to be tested green were immediately dressed to $1\frac{1}{2}$ in. square and tested as quickly as possible. Those to be tested seasoned were left rough until they reached the desired moisture content, then were dressed to the same size and tested.

TESTS AND TESTING METHODS

Strength tests reported here are limited to compression parallel to the grain and bending, together with determination of specific gravity, percentage of sapwood and summerwood, rate of growth, and moisture content.

The fiber saturation point was investigated by a modification of the electro-conductivity method introduced by Myer and Rees(2). Extensive studies of resin content, resin ducts, ray cells, fiber dimensions and conformation, absolute density of wood substance and other items relating to the minute structure were made and reported in detail by Berkley(1).

The testing methods used conformed in a general way to the Society's Standard Methods of Testing Small Clear Specimens of Timber (D 143-27).[1] The following exceptions were made: Test specimens were $1\frac{1}{2}$ in. square instead of 2 in. This allowed more tests per bolt and a better division of the strength zones over the cross-section. The specified proportions of the test specimen were retained—compression specimens were four times as long as wide and bending specimens were tested over a span fourteen times the depth.

Load-deformation curves were plotted for all specimens except for certain series from trees Nos. 1 and 5. This procedure made possible a closer analysis of the relations between elastic properties and maximum strength than was possible if A.S.T.M. Methods D 143 had been followed. Moisture samples were taken from unstressed material between test specimens instead of from the fractured specimen, thus producing more concordant results in green material containing much moisture.

Deformations in compression tests were measured over the entire length instead of over a gage length marked on the specimen; values of proportional limit and modulus of elasticity in compression are, therefore, not directly comparable with values obtained under A.S.T.M. Methods. Several hundred tests[2] of short columns of different lengths, in which deformations were measured over the full length and over several marked gage lengths simultaneously, indicated clearly that (1) unit deformations measured over the whole length were quite as consistent as when measured over a marked gage length, but were consistently larger, (2) when measured over gage lengths approaching the full length of specimen, the values approached those measured between testing heads, and (3) deformations in inches per inch of specimen were considerably greater near the ends than near the center of specimens. Moreover, the relations between maximum strength, proportional limit and modulus of elasticity were more consistent when measured over the full length of specimen than when the standard procedure was followed.

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[1] 1933 Book of A.S.T.M. Standards, Part II, p. 408.

[2] Tests by A. W. Brust, S. W. Jens, C. E. Abbey and A. E. Biermann, Department of Civil Engineering, Washington University (1933–1934). (Unpublished.)

The specific gravity reported is based on the volume at time of test and the oven-dry weight. Consequently, the specific gravity as reported for green material is consistently lower than for the same material tested dry, due to shrinkage. Volumes for specific gravity determinations were obtained by the immersion method. Dry sapwood specimens were found to absorb from 3 to 10 per cent of their dry weight while being weighed under water, whereas heartwood absorbed less than 1 per cent. Dipping specimens in hot paraffin before immersion increased the volume an indeterminate amount averaging between 4 and 5 per cent. The error in volume due to either the absorption or the paraffin caused the calculated specific gravity to be in error by 5 to 15 per cent. To avoid these errors, dry sapwood pieces were weighed in air and under water without coating with paraffin and immediately reweighed in air and a correction was made for the absorbed water in computing the volume. Specific gravities in this paper do take account of resin and other soluble materials.

STRUCTURE OF PINE STEM

The pine stem, or xylem, is made up of fiber-like cells known as tracheids, which in straight-grained trees lie parallel to the axis of the tree, and in spiral-grained trees lie at an angle to the axis. In the first rings around the pith the fibers are very short, being less than a millimeter in average length, and very thin walled, with few thick-walled summerwood cells. As the successive rings are laid down the fibers increase in length, rapidly at first and then more slowly, reaching an average length of from 4 to 5.5 mm. between about the fortieth and one hundredth rings. As the length increases the walls in general become thicker and the percentage of summerwood increases, thus increasing the density. The last rings formed in an old tree (after the age of 80 to 100 yr.), however, contain relatively long tracheids but less summerwood. Thus the density again decreases towards the bark. The maximum strength for any cross-section will be found in the long and straight-fibered, high-density material, and its position as to distance from the pith will depend upon the individual tree.

There are two extreme types of tracheids based on the structure of the cell walls, with intergrading forms between these two. The cell walls of the most common type are made up of concentric layers or laminae and have been defined as "concentrimural" tracheids by Berkley(1) as follows: "Concentrimural tracheids may be defined as cells usually without interstitial spaces, thin- or thick-walled, the secondary thickening and the middle lamella of which are made up of layers or laminae in the form of concentric cylinders." When straight and uniform they form a compact structure, but crooked or deformed tracheids of this type may contain pronounced interstitial spaces. The second type has been defined as "torquimural" tracheids by Berkley(1) or "tracheids with or without interstitial spaces, moderately to very dense, the secondary wall of the individual cell differentiated from the primary wall by its radial bands or laminae which lie in a spiral around the cell." When these cells occur in mass they are known as "compression wood" or "Rothholz." Torquimural cells usually have pronounced interstitial spaces and are often crooked and irregular in shape

TABLE II.—RESULTS OF COMPRESSION TESTS.

Tree and Bolt	Species	Number of Tests	Moisture Content, per cent	Specific Gravity	Percentage of Summer-wood	Rings per inch	Proportional Limit, lb. per sq. in.	Compressive Strength, lb. per sq. in.	Ratio, Proportional Limit to Compressive Strength	Modulus of Elasticity, lb. per sq. in.
GREEN SOUTHERN YELLOW PINE										
5-a	Loblolly	58	....	....	54	7.1	....	3 931	....	....
5-b		53	....	0.549	49	7.8	2681	3 890	0.689	1 029 000
5-i		49	....	0.459	29	9.4	2320	3 485	0.666	989 000
5-q		41	....	0.426	24	8.8	1886	2 840	0.664	743 000
7-a and b		29	....	0.468	43	5.4	2466	3 365	0.734	633 300
7-g and h		24	....	0.447	32	5.2	2379	3 214	0.741	702 000
7-m and n		22	....	0.434	24	5.8	2286	3 062	0.746	667 000
3-a and b	Shortleaf	29	....	0.590	39	11.2	3641	4 823	0.758	1 340 000
3-c and d		23	....	0.540	35	12.0	3343	4 323	0.775	1 143 000
3-e and f		23	....	0.515	34	10.5	3010	3 920	0.766	1 004 000
3-g and h		22	....	0.505	31	12.0	2738	3 607	0.763	920 000
4-a	Longleaf	22	....	0.565	38	19.5	3116	3 938	0.791	1 166 000
4-b		21	....	0.540	36	20.0	3078	3 895	0.784	1 142 000
4-c		20	....	0.572	36	21.0	3094	4 144	0.745	1 133 000
4-d		20	....	0.527	31	20.0	3011	3 872	0.778	1 098 000
4-e		16	....	0.540	42	20.5	2679	3 534	0.711	932 000
4-f		15	....	0.555	40	20.0	2930	3 736	0.788	1 032 000
4-g		14	....	0.545	34	21.0	2794	3 608	0.777	983 000
4-h		14	....	0.521	29	21.0	2348	3 272	0.722	925 000
6-a and b		24	....	0.557	43	24.8	3401	4 491	0.757	1 305 000
6-e and f		19	....	0.508	38	24	2624	3 674	0.714	980 000
6-i and j		17	....	0.487	31	21	2419	3 330	0.727	829 000
SEASONED SOUTHERN YELLOW PINE										
1-a	Loblolly	25	6.6	0.589	57	7.7	6445	10 270	0.613	1 595 000
1-d		22	7.0	0.500	34	7.5	....	9 400	....	....
1-h		28	7.9	0.473	29	6.9	....	8 385	....	....
2-a		28	8.0	0.596	52	5.4	7255	11 150	0.635	1 721 000
2-d		24	6.2	0.508	43	4.5	5750	9 025	0.622	1 400 000
2-h		24	7.7	0.495	30	4.0	5300	8 390	0.627	1 240 000
5-a		30	13.5	0.591	56	7.2	3639	6 057	0.601	1 165 000
5-i		25	12.6	0.478	36	8.9	4036	6 036	0.669	1 137 000
5-q		22	12.2	0.449	25	8.3	3604	5 568	0.647	1 154 000
5-b		48	13.5	....	50	8.5	....	6 203	....	....
5-i		44	12.5	....	30	10.0	....	5 733	....	....
5-q		36	12.0	....	23	....	....	5 117	....	....
5-b		25	7 to 9	0.610	..	....	....	9 664	....	....
5-i		25	7 to 9	0.497	..	....	....	8 236	....	....
5-q		34	7 to 9	0.455	..	....	....	6 724	....	....
5-b		25	0.0	0.629	51	....	....	12 815	....	....
5-i		25	0.0	0.528	26	....	....	11 124	....	....
5-q		20	0.0	0.496	24	....	....	9 255	....	....
3-a and b	Shortleaf	26	8.4	0.674	42	11.7	7143	10 595	0.666	2 053 000
3-c and d		24	8.9	0.568	36	13.0	6940	10 075	0.685	1 926 000
3-e and f		24	10.0	0.570	30	12.3	5707	8 248	0.683	1 630 000
3-g and h		20	8.9	0.518	30	13.4	6042	8 442	0.706	1 564 000

and length. They have a greater longitudinal shrinkage than the concentrimural cells, thus causing extensive warping and shaking in small timbers. In large timbers the torquimural cells often rupture, forming cross breaks.

Other elements of the pine stem are the parenchyma cells which are common around the resin ducts and often occur in isolated groups particularly in the springwood, and the wood rays. The resin ducts are canal-like, intercellular spaces running parallel with the tracheids, and laterally in the fusiform rays. The longitudinal resin ducts and the wood rays were very numerous in the first rings, decreasing in number toward the periphery but increasing in diameter in the same direction. Numerous resin ducts, together with the thin-walled parenchyma cells around them, decreased the specific gravity. The wood rays, especially the fusiform rays, interrupted the tracheids at the point where they passed, causing the fibers to be slightly crooked and smaller in diameter. (See Berkley for full description(1).)

TABLE III.—RESULTS OF BENDING TESTS.

Tree and Bolt	Species	Number of Tests	Moisture Content, per cent	Specific Gravity	Percentage of Summerwood	Rings per inch	Proportional Limit, lb. per sq. in.	Modulus of Rupture, lb. per sq. in.	Ratio, Proportional Limit to Modulus of Rupture	Modulus of Elasticity, lb. per sq. in.
GREEN SOUTHERN YELLOW PINE										
5-a	Loblolly	57	....	0.549	50	8.0	3 630	8 138	0.446	1 336 000
5-i		48	....	0.459	48	8.8	3 428	7 187	0.447	1 304 000
5-q		41	....	0.426	25	8.8	3 002	6 221	0.483	1 172 000
7-a and b		28	....	0.491	43	5.4	3 108	7 077	0.439	1 137 000
7-g and h		24	....	0.463	34	5.0	3 150	6 374	0.495	1 034 000
7-m and n		19	....	0.431	31	4.5	2 871	6 115	0.469	1 053 000
3-a and b	Shortleaf	28	....	0.588	47	12.0	4 067	9 555	0.425	1 805 000
3-c and d		22	....	0.540	36	11.8	3 524	9 016	0.398	1 640 000
3-e and f		21	....	0.515	30	12.7	2 986	8 434	0.353	1 890 000
3-g and h		23	....	0.484	33	12.3	3 592	7 680	0.470	1 555 000
4-a	Longleaf	20	....	0.565	35	17.8	3 528	7 968	0.451	1 482 000
4-b		18	....	0.547	34	21.0	3 058	8 530	0.363	1 513 000
4-c		20	....	0.572	36	20.0	2 945	7 822	0.374	1 661 000
4-d		20	....	0.527	30	20.0	2 960	7 893	0.373	1 668 000
4-e		15	....	0.540	39	21.0	3 195	7 933	0.405	1 516 000
4-f		14	....	0.555	41	21.0	2 926	7 882	0.372	1 612 000
4-g		14	....	0.545	39	20.0	2 874	7 796	0.379	1 420 000
4-h		13	....	0.521	38	20.0	3 113	7 342	0.428	1 449 000
6-a and b	Longleaf	22	....	0.558	46	26	4 103	8 885	0.462	1 776 000
6-e and f		19	....	0.524	42	22	3 610	8 420	0.430	1 572 000
6-i and j		16	....	0.490	42	24	3 124	7 328	0.424	1 324 000
SEASONED SOUTHERN YELLOW PINE										
1-a	Loblolly	23	6.6	0.587	56	6.7	8 595	16 165	0.530	1 834 000
1-d		22	7.0	0.500	37	7.9	....	15 750	....	....
1-h		20	7.4	0.473	28	6.8	....	14 610	....	....
2-a		26	8.0	0.596	50	5.3	9 736	18 230	0.534	2 111 000
2-d		24	6.2	0.508	41	4.8	7 870	14 110	0.555	1 664 000
2-h		24	7.7	0.495	29	4.0	8 298	13 400	0.598	1 636 000
3-a and b	Shortleaf	19	8.4	0.670	41	11.7	10 710	17 850	0.595	2 368 000
3-c and d		23	8.9	0.593	37	13.0	9 502	16 495	0.574	2 263 000
3-e and f		19	10.0	0.570	29	11.7	6 730	13 020	0.524	1 967 000
3-g and h		20	8.9	0.518	25	13.0	8 128	13 770	0.588	1 875 000
5-a	Loblolly	27	13.5	0.591	52	8.1	5 325	12 275	0.434	1 556 000
5-i		22	12.6	0.478	36	9.0	6 388	11 580	0.543	1 611 000
5-q		20	12.2	0.449	29	8.6	5 705	10 090	0.565	1 416 000

RESULTS OF STRENGTH TESTS

The compression and bending strengths, proportional limits and moduli of elasticity of some 1700 clear specimens taken from the seven trees, together with their moisture content, specific gravity, percentage of summerwood and rate of growth are shown in Tables II and III. The proportional limit is taken as the stress at which deformation first ceases to be proportional to load and therefore marks the first deviation of the stress-strain diagram from a straight line. The tables show only the average values of all the specimens in a bolt, or, in some cases, two bolts. All of these tests were made under identical testing conditions. (Nearly a thousand other tests were made on material from the same trees to study the effect of variations in testing procedure, such as variation in specimen dimensions, seasoning conditions, and methods of measuring deformations.)

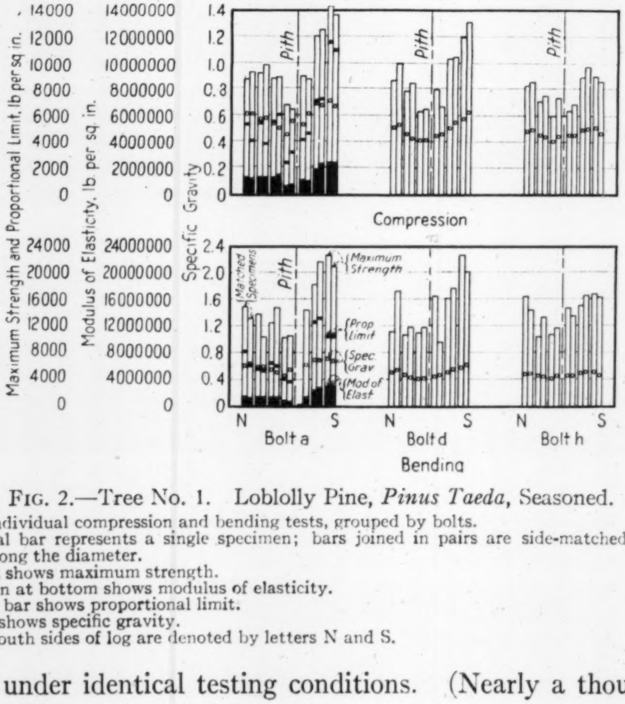

FIG. 2.—Tree No. 1. Loblolly Pine, *Pinus Taeda*, Seasoned.

Results of individual compression and bending tests, grouped by bolts.

Each vertical bar represents a single specimen; bars joined in pairs are side-matched specimens and represent 2 in. along the diameter.

Total height shows maximum strength.

Black portion at bottom shows modulus of elasticity.

Heavy cross bar shows proportional limit.

Small circle shows specific gravity.

North and south sides of log are denoted by letters N and S.

One series of these tests through the north-south diameter of three bolts of each of the seven trees is presented in Figs. 2 to 15. The "bar graphs" for each tree are designed to show the variations in specific gravity, strength, stiffness, and proportional limit in bending and compression for the individual tests.

A comparison of the heights of the bars in each bolt shows the variations from bark to bark across the diameter of the tree. Comparison of the bolts in each figure shows the differences in strength due to height in

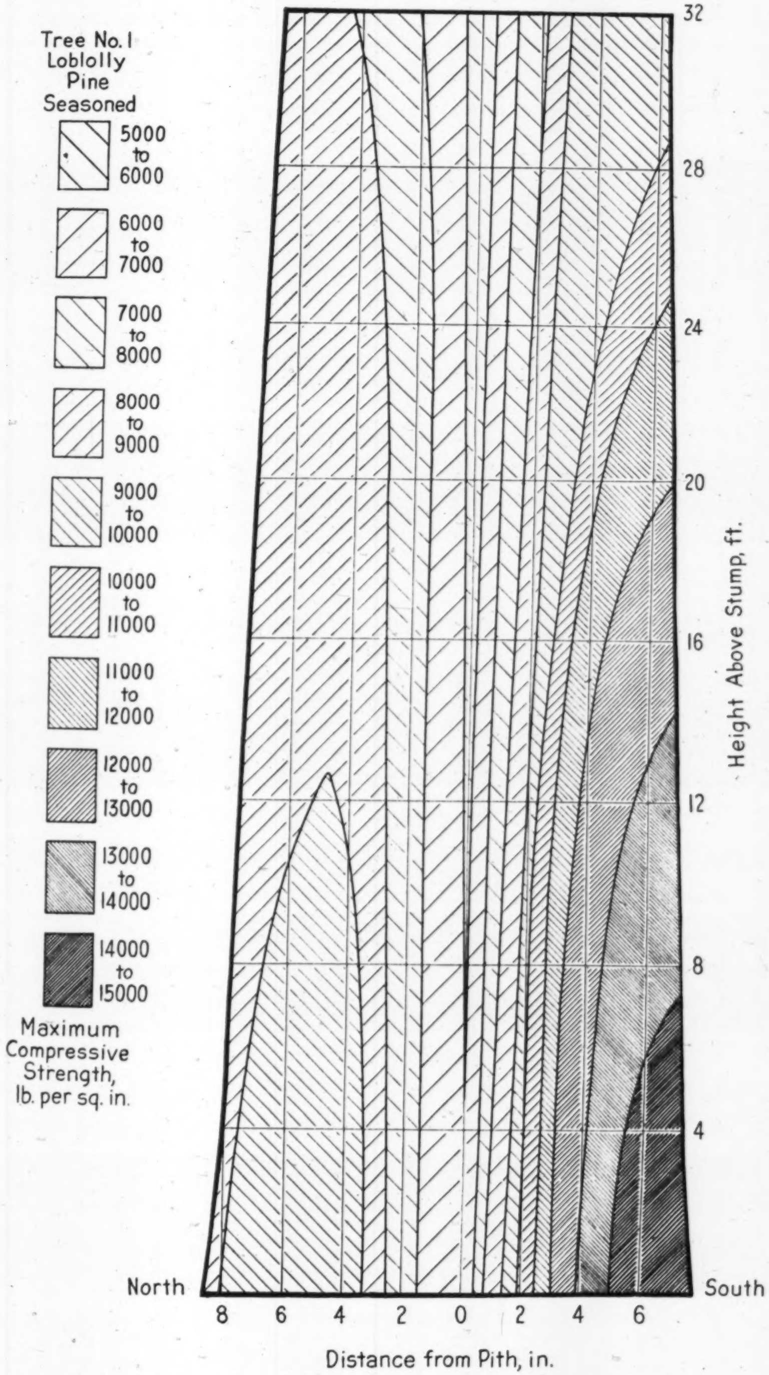

FIG. 3.—Tree No. 1. Loblolly Pine, *Pinus Taeda*, Seasoned.
Relation of strength in compression parallel to the grain to position in the tree.

the tree, but these relations are more readily observed in the tree diagrams constructed from the compression tests represented in the bar graphs showing the distribution of strength in the tree.

In the tree diagrams the division lines between strength areas were obtained by interpolating between successive test specimens, small local variations being disregarded. In most of these diagrams (particularly Fig. 3) the north and south sides of the trees show different strength values; therefore, diagrams plotted on a plane through the east-west diameter may be expected to differ from those shown. If, however, the bending strength or the elastic properties of these same specimens were plotted in the same manner the diagrams would be very similar to the compressive strength diagrams, as indicated by the bar graphs.

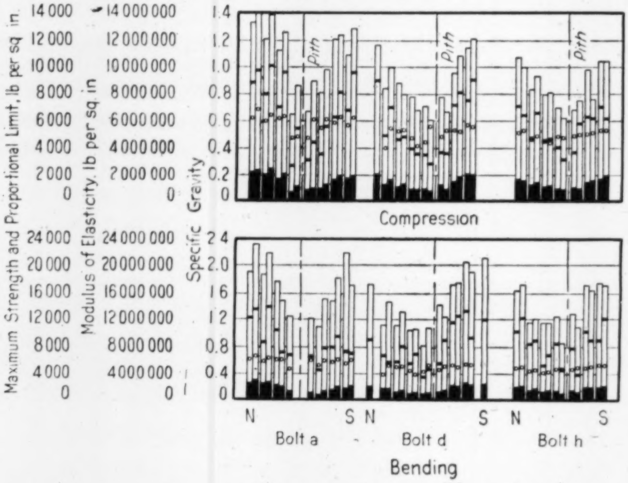

FIG. 4.—Tree No. 2. Loblolly Pine, *Pinus Taeda*, Seasoned.

Results of individual compression and bending tests, grouped by bolts.
Each vertical bar represents a single test; bars joined in pairs are side-matched specimens and represent 2 in. along the diameter.
Total height shows maximum strength.
Black portion at bottom shows modulus of elasticity.
Heavy cross bar shows proportional limit.
Small circle shows specific gravity.
North and south sides of log are denoted by letters N and S.

Figure 2 (tree No. 1, loblolly pine, see Table I) shows the results of compression and bending tests of seasoned wood from bolts a, d, and h. The growth rate was extremely rapid for the first seven years, but much slower thereafter. The wide ringed material near the pith consisted largely of short, thin-walled cells which were crooked and deformed near the ends, with numerous interstitial spaces. Farther from the pith, the narrower ringed south side was materially stronger than the north side (see Fig. 3). The difference in strength was due to numerous torquimural cells appearing as compression wood on the north side, together with numerous large rays

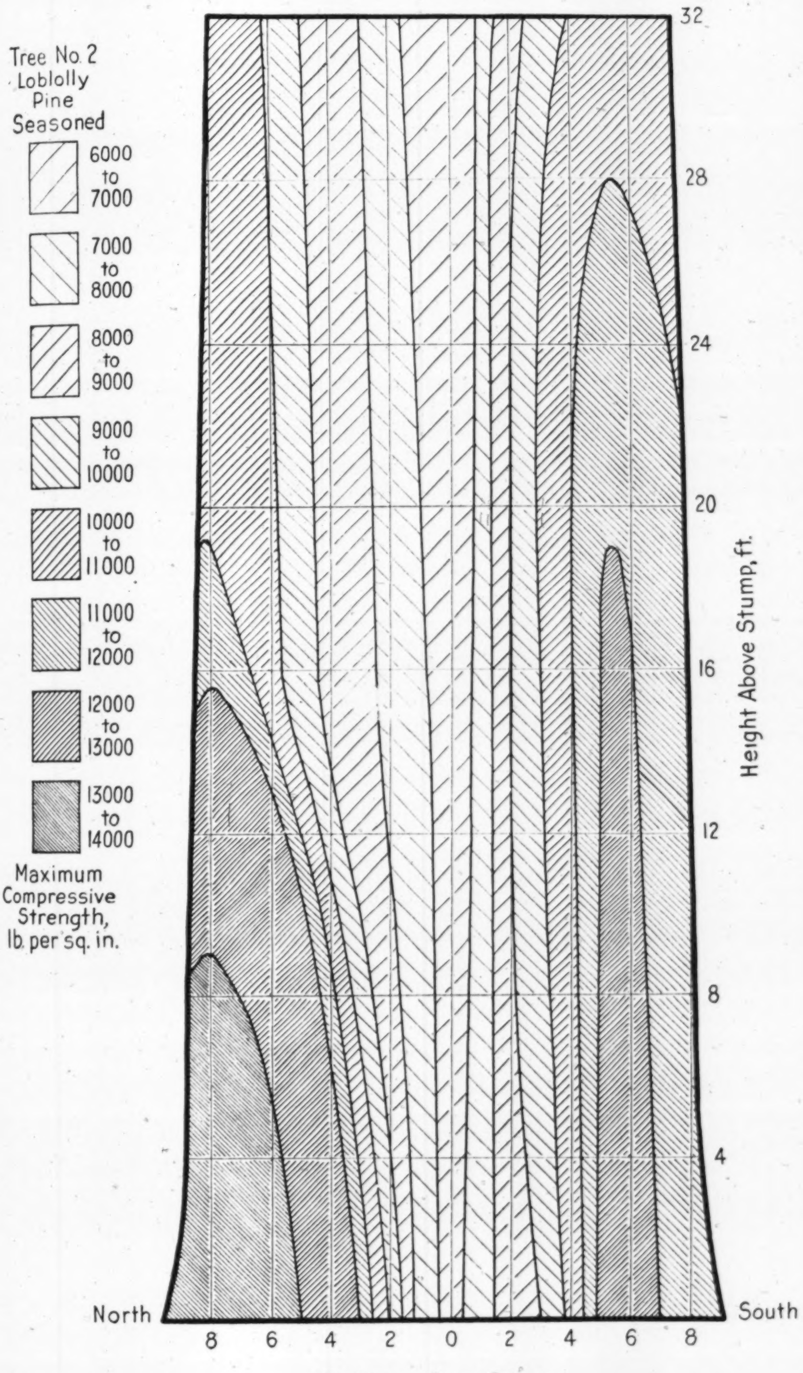

FIG. 5.—Tree No. 2. Loblolly Pine, *Pinus Taeda*, Seasoned.
Relation of strength in compression parallel to the grain to position in the tree.

and crooked tracheids, and not to the width of rings or the density. There were few torquimural cells on the south side and no distinct compression wood rings. The strength in compression and bending doubled from the pith to the bark; the proportional limit and modulus of elasticity varied even more. The stronger wood on the south side contained less summerwood than that on the north side and was stronger, stiffer and more elastic in terms of its density than either the north side or the wide ringed material near the pith. When only the average values of the entire cross-section are shown as in Tables II and III, these irregularities are not apparent.

Figure 2 shows a close coordination between compressive and bending strength of specimens from the same stick. In general, a specimen strong in compression was also strong in bending, but, as will be seen by comparing with other trees, the ratio between compression and bending strength differs greatly in green and seasoned wood and in wood containing different fiber structures.

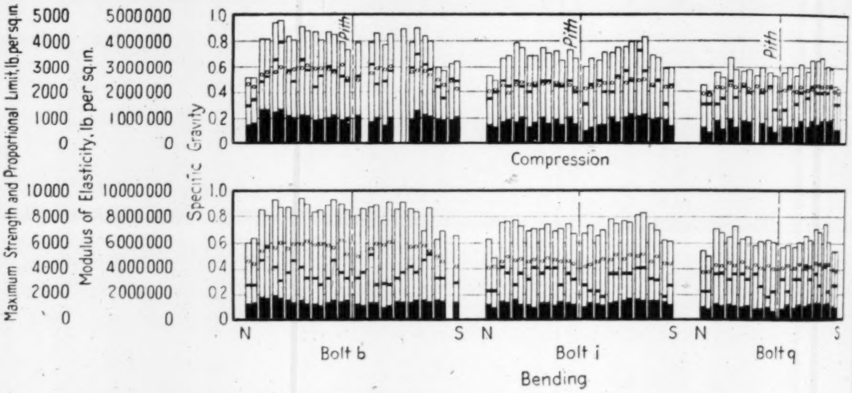

FIG. 6.—Tree No. 5. Loblolly Pine, *Pinus Taeda*, Green.

Results of individual compression and bending tests, grouped by bolts.
Each vertical bar represents a single specimen; bars joined in pairs are side-matched specimens and represent 2 in. along the diameter.
Total height shows maximum strength.
Black portion at bottom shows modulus of elasticity.
Heavy cross bar shows proportional limit.
Small circle shows specific gravity.
North and south sides of log are denoted by letters N and S.

Tree No. 1 was straight but leaned slightly with the dominant branches on the leaning side. The average strength was high but much of the wood warped badly in seasoning.

Tree No. 2, loblolly pine, had about the same age, diameter, and growth characteristics as tree No. 1 (see Table I). Strength again doubled from pith to bark but the increase in all directions from the pith was more nearly uniform and changes in growth rate were less abrupt than in tree No. 1. As seen in Fig. 4, there was very little change in density over the

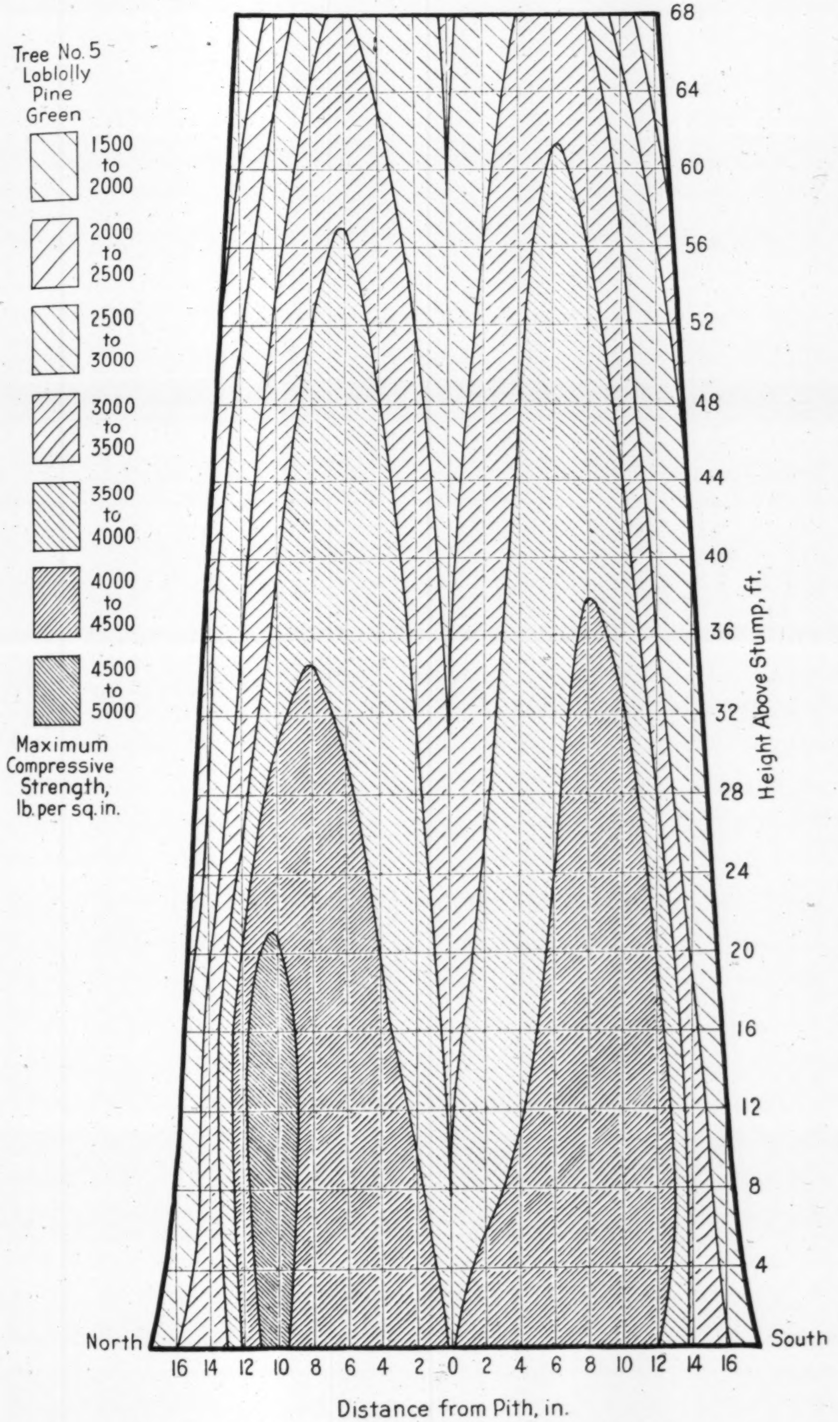

FIG. 7.—Tree No. 5. Loblolly Pine, *Pinus Taeda*, Green.
Relation of strength in compression parallel to the grain to position in the tree.

cross-section. The modulus of elasticity followed the maximum strength very closely but the proportional limit was very irregular.

The wood from tree No. 2 was extremely brash. Most of the bending specimens broke into two parts at the first sign of failure. This extreme brashness was caused partly by a large percentage of torquimural cells and partly by large, thin-walled springwood cells with crooked ends. This tree was also leaning slightly but there were few, if any, definite compression wood rings. Torquimural cells were numerous on the east and south sides and were found to some extent on all sides and at all heights in the tree. Resin ducts and wood rays were large and numerous in the wide-ringed heart wood and in local areas of sapwood. Wood from tree No. 2 warped more than that from any other tree studied.

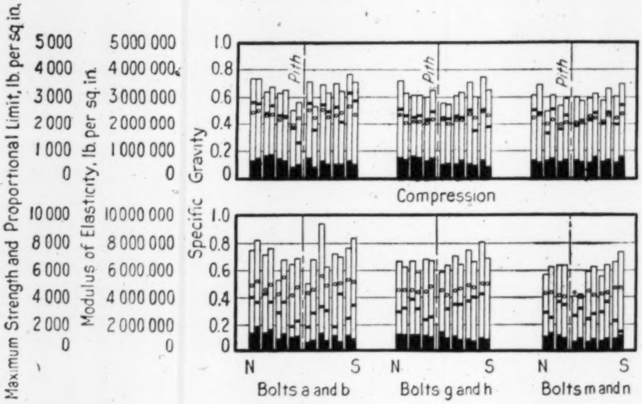

FIG. 8.—Tree No. 7. Loblolly Pine, *Pinus Taeda*, Green.

Results of individual compression and bending tests, grouped by bolts.

Each vertical bar represents a single specimen; bars joined in pairs are end-matched specimens and represent 2 in. along the diameter.

Total height shows maximum strength.

Black portion at bottom shows modulus of elasticity.

Heavy cross bar shows proportional limit.

Small circle shows specific gravity.

North and south sides of log are denoted by letters N and S.

Tree No. 5 was a particularly fine example of old, straight, uniform-ringed loblolly pine. This tree grew uniformly for the first eighty years at the rate of five to eight rings per inch after which the growth rate decreased sharply to between fifteen and thirty rings per inch. The strength increased uniformly from the pith to a maximum near the point where there was a sharp decrease in growth rate, beyond which the strength dropped suddenly to a minimum (see Fig. 6). The coordination between density, strength, stiffness and proportional limit was exceptionally close for any given bolt in this tree, both in the green and seasoned specimens. The tree contained only a few scattered torquimural cells, but the butt of the tree and to some extent the middle and top contained crooked and deformed tracheids.

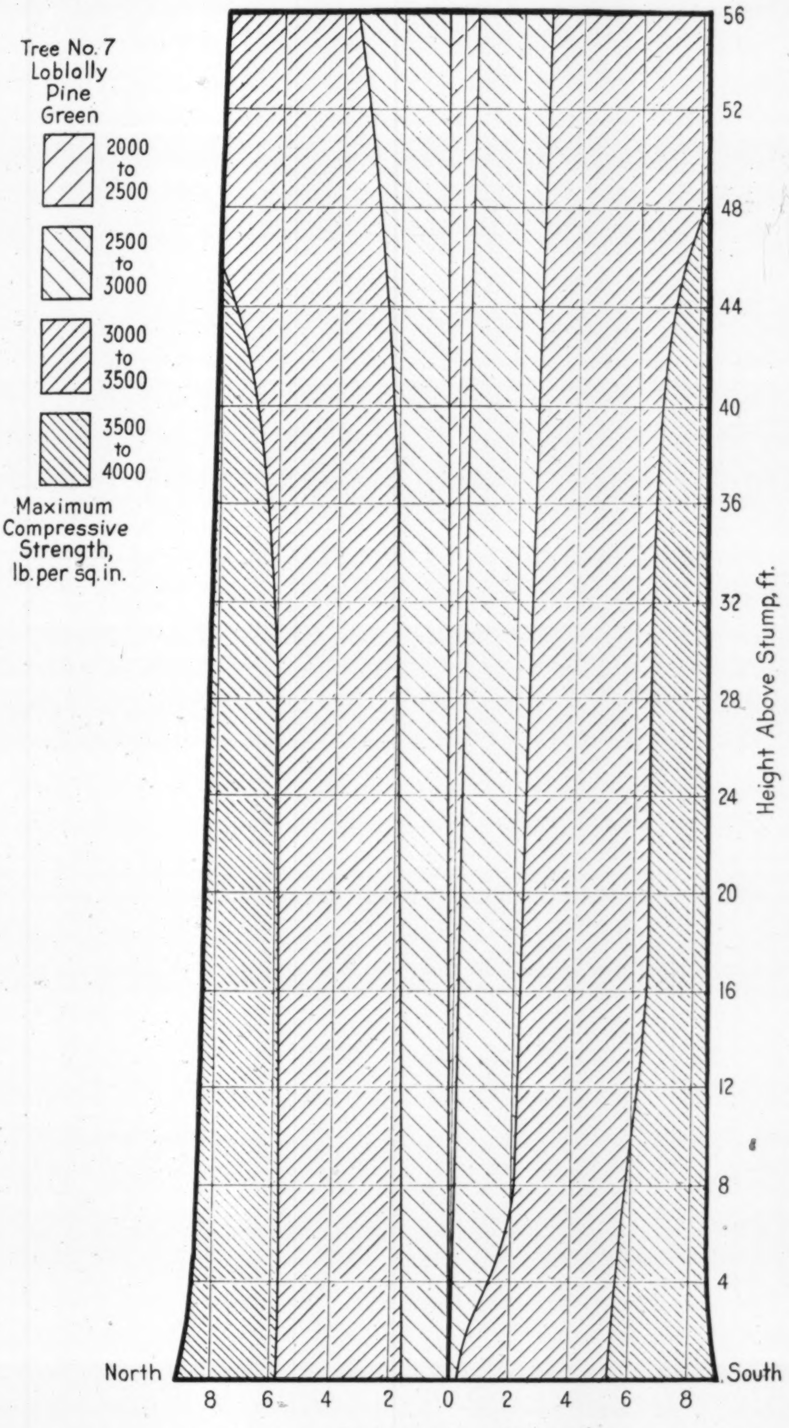

FIG. 9.—Tree No. 7. Loblolly Pine, *Pinus Taeda*, Green.
Relation of strength in compression parallel to the grain to position in the tree.

Practically all of the wood from tree No. 5 would grade as dense, select southern yellow pine (Southern Pine Association). When tested green, only four tests per hundred showed a modulus of elasticity in bending equal to or greater than 1,600,000 lb. per sq. in. and the modulus of elasticity in compression was less than 1,000,000 lb. per sq. in. in more than 50 per cent of the tests.

Tree No. 7, loblolly pine, grew somewhat more rapidly than tree No. 5 but was otherwise very similar in density, strength and stiffness properties and distribution to the first sixty rings of tree No. 5 (see Figs. 8 and 9).

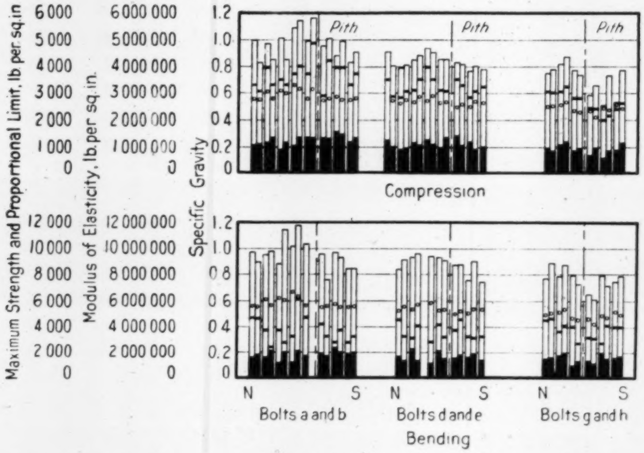

FIG. 10.—Tree No. 3. Shortleaf Pine, *Pinus Echinata*, Green.

Results of individual compression and bending tests, grouped by bolts.

Each vertical bar represents a single test specimen; bars joined in pairs are end-matched specimens and represent 2 in. along the diameter.

Total height shows maximum strength.

Black portion at bottom shows modulus of elasticity.

Heavy cross bar shows proportional limit.

Small circle shows specific gravity.

North and south sides of log are denoted by letters N and S.

Tree No. 3, shortleaf pine, grew on a steep northwest slope. For the first 20 ft. above the stump, the pith was about 2 in. off center toward the south but was nearly centered above that point. The wide and narrow ringed wood from the north and south sides, respectively, showed little variation in strength, stiffness and density which were high and uniform throughout the cross-section, but end-matched specimens from the same sticks in the two bottom bolts showed rather large differences (see Fig. 10).

In terms of density and maximum strength, the proportional limit in compression was very high while in bending it was unusually low. In bending, the ratio of proportional limit to maximum strength was materially greater in seasoned than in green wood.

Tree No. 4, longleaf pine, grew on a steep northwest slope in a dense original stand, recently cut over (see Table I). The ring structure indicated

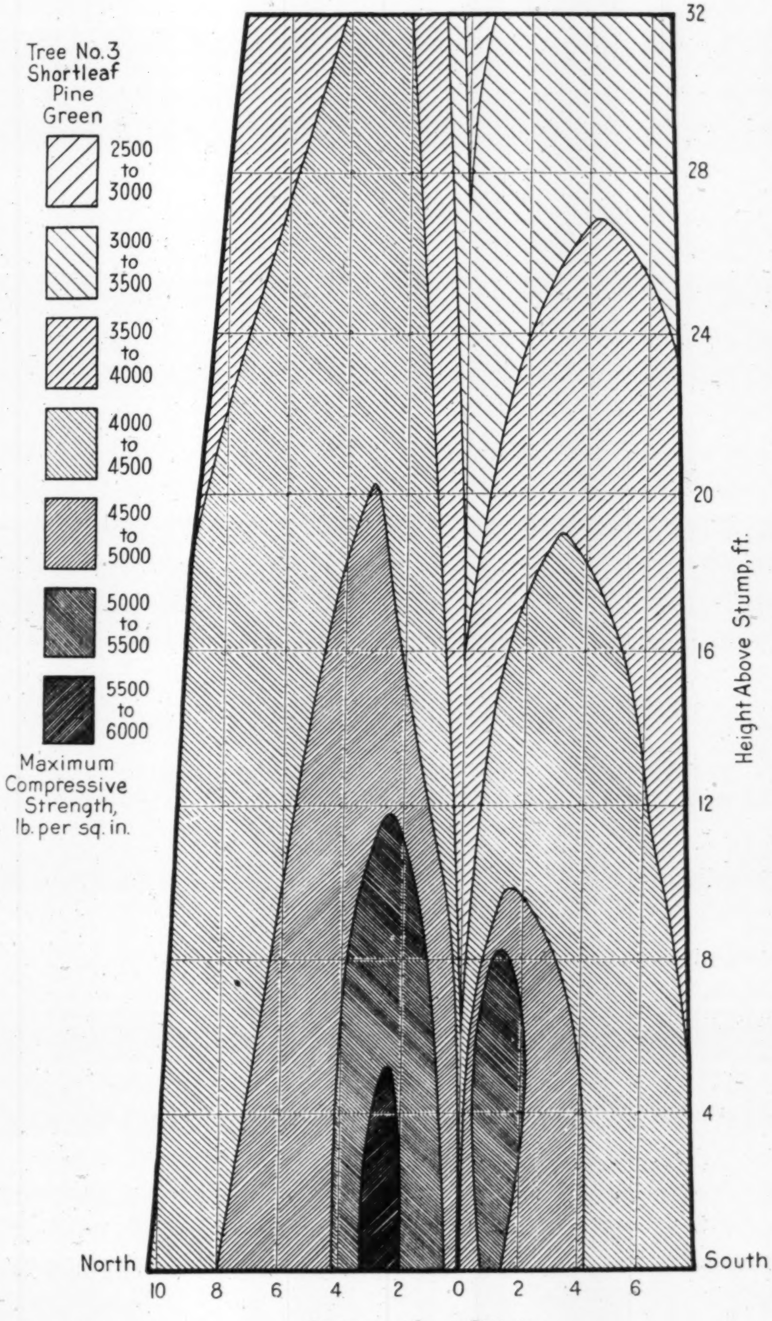

FIG. 11.—Tree No. 3. Shortleaf Pine, *Pinus Echinata*, Green.
Relation of strength in compression parallel to the grain to position in the tree.

alternating periods of very slow and somewhat faster growth throughout the life of the tree. The wide fluctuations in density were only poorly reflected by the strength properties but they were due in large measure to the very irregular distribution of resin content which was relatively high in all parts of the tree. Occasional rings of compression wood and areas of crooked tracheids also affected the strength-density relations. Figure 13 shows a peculiar distribution of strength in the tree. The pith was more crooked than the log itself and the strength shifted from side to side with the curves in the pith. Strength variations of side-matched specimens are due largely to areas of local cross grain.

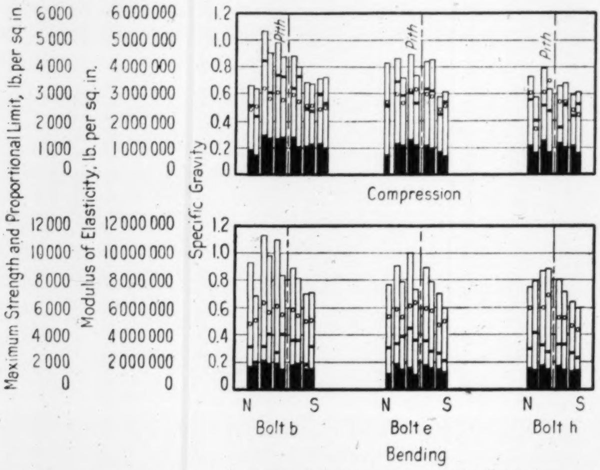

FIG. 12.—Tree No. 4. Longleaf Pine, *Pinus Palustris*, Green.

Results of individual compression and bending tests, grouped by bolts.
Each vertical bar represents a single specimen; bars joined in pairs are side-matched specimens and represent 2 in. along the diameter.
Total height shows maximum strength.
Black portion at bottom shows modulus of elasticity.
Heavy cross bar shows proportional limit.
Small circle shows specific gravity.
North and south sides of log are denoted by letters N and S.

Tree No. 6, longleaf pine, like tree No. 4 showed a marked decrease in strength from the inner heartwood to the bark with a corresponding decrease in density in the same direction. This tree was from a cutover stand. The growth rate more than doubled after the surrounding trees had been cut but the tree was past the age at which maximum density and strength are usually found.

Figures 16 and 17 show the densities of single green specimens plotted against strength in compression and bending respectively. The loblolly pine was low in strength and density when compared with the other two species; for densities below 0.55, however, loblolly pine was equal in strength to shortleaf and equal or superior to longleaf pine of the same density. The densities shown in these figures disregard resin content; if the resin

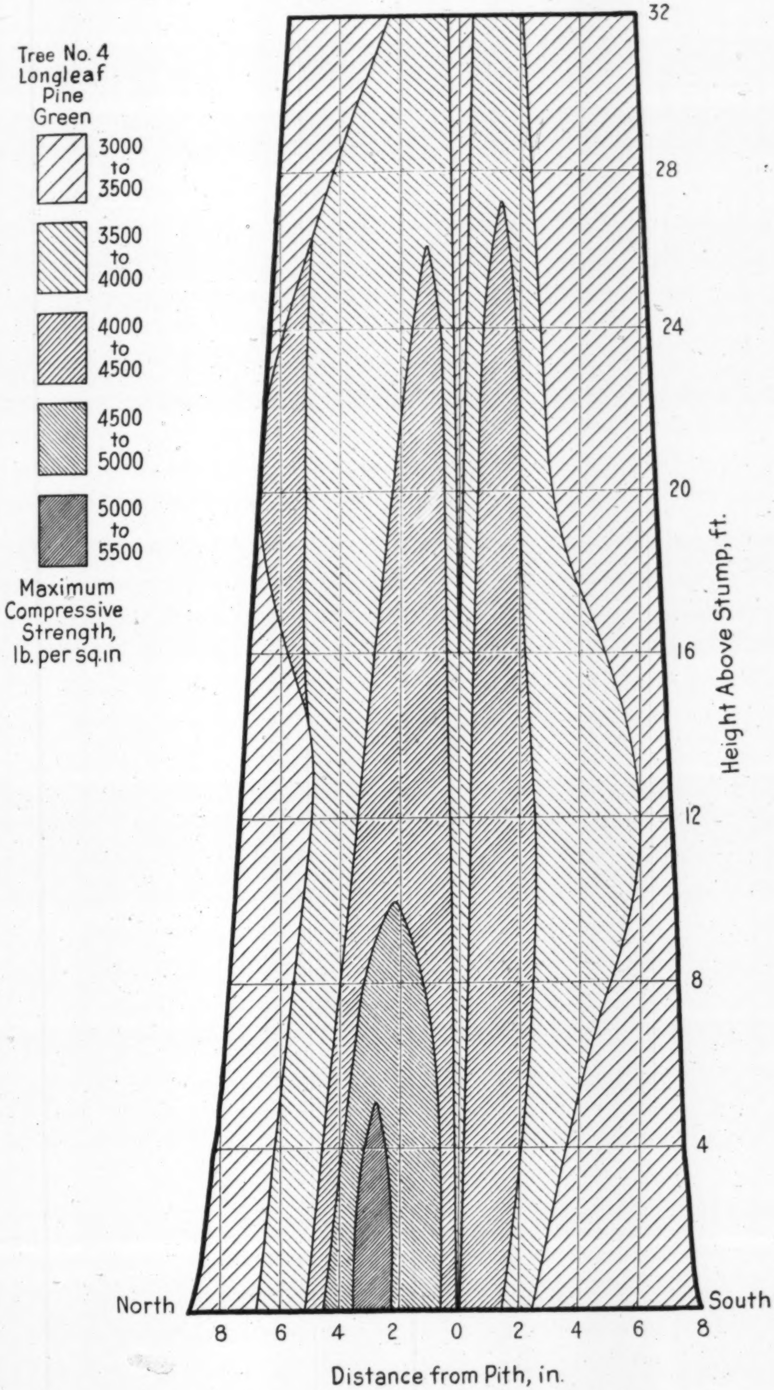

FIG. 13.—Tree No. 4. Longleaf Pine, *Pinus Palustris*, Green.
Relation of strength in compression parallel to the grain to position in the tree.

is eliminated, longleaf pine slightly surpassed the other species because of its slightly thicker cell walls. The resin content averaged 2.3, 2.5 and 5.6 per cent for loblolly, shortleaf and longleaf pines, respectively (Berkley(1)).

Note that the deviation becomes larger as the densities increase. The extreme variations are partially due to resin content but variations of 2000 lb. per sq. in. in compression and twice as much in bending for a given density remain after the resin is eliminated.

Although the average compressive strength and density of the 650 tests represented in Fig. 16 agree within 5 per cent with similar averages reported by Newlin and Wilson(3) in 1917, the *relation* between strength and density does not agree with their general equation reported in 1919(4). The average increase in strength for 0.10 increase in density was 950 lb. per sq. in. when computed by the method of least squares. The corresponding increase for those specimens containing only straight, concentrimural fibers was 1350 lb. per sq. in. The average increase reported by Newlin and Wilson in 1919(4) for *all* woods was 690 lb. per sq. in. The differences in these values are due to the varying proportion of different types of cell structure in the material tested.

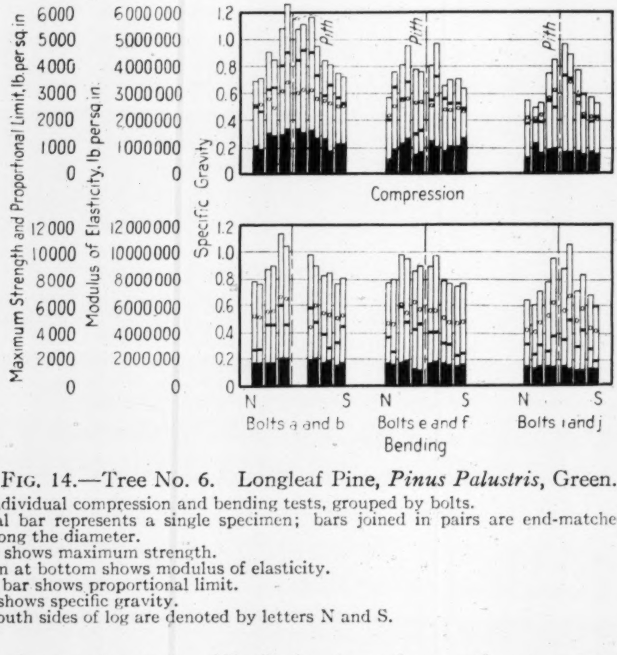

FIG. 14.—Tree No. 6. Longleaf Pine, *Pinus Palustris*, Green.

Results of individual compression and bending tests, grouped by bolts.
Each vertical bar represents a single specimen; bars joined in pairs are end-matched specimens and represent 2 in. along the diameter.
Total height shows maximum strength.
Black portion at bottom shows modulus of elasticity.
Heavy cross bar shows proportional limit.
Small circle shows specific gravity.
North and south sides of log are denoted by letters N and S.

If only the averages of species had been plotted, the data in Figs. 16 and 17 would have been represented by only three points in each figure, that is, one each for loblolly, shortleaf and longleaf pine. The range in density would have been from 0.50 to 0.55 instead of from 0.36 to 0.76 as

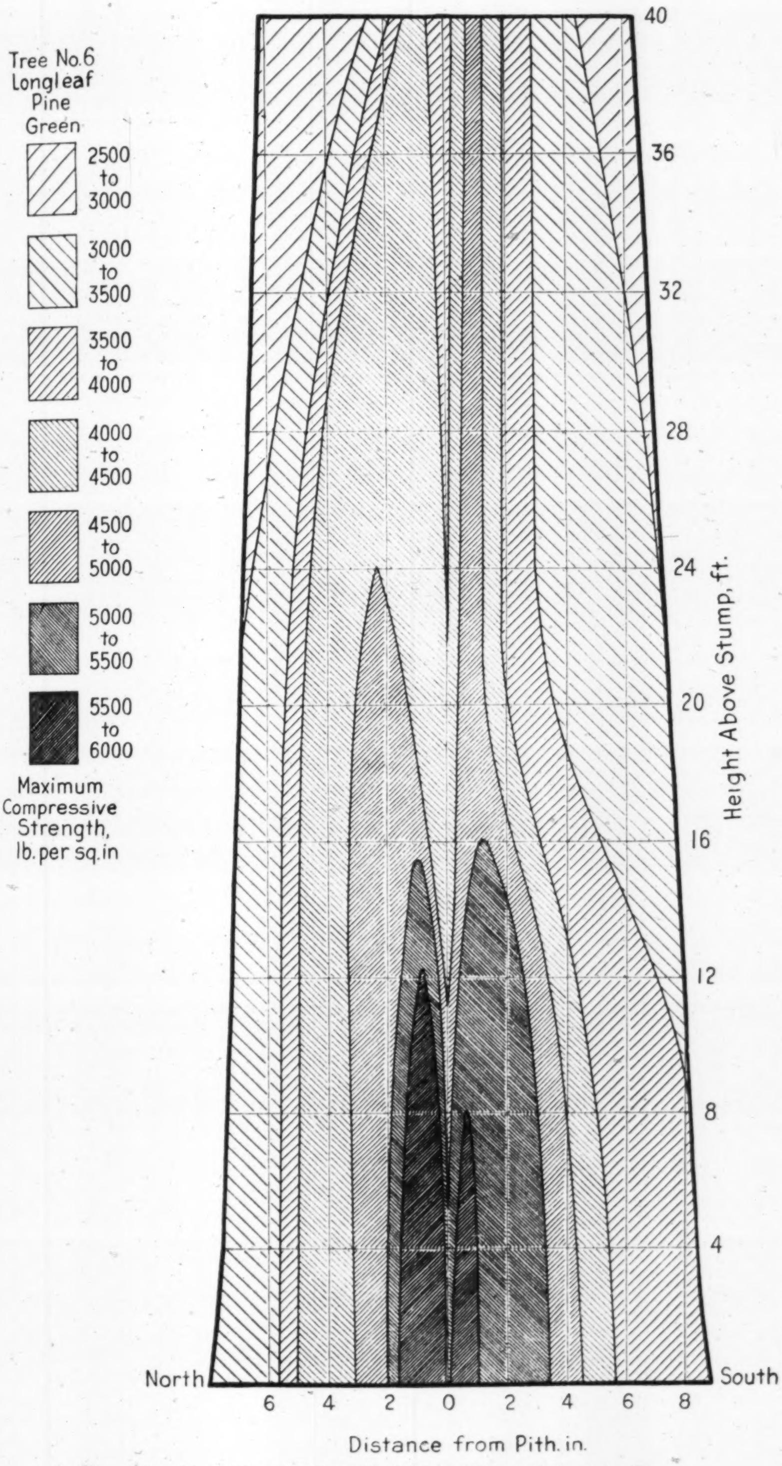

FIG. 15.—Tree No. 6. Longleaf Pine, *Pinus Palustris*, Green.
Relation of strength in compression parallel to the grain to position in the tree.

shown, and the range of compressive strength would have been from 3580 to 4390 lb. per sq. in. instead of from 2000 to 6300 lb. per sq. in. This illustrates the great extent to which variability in strength and density is masked by considering only averages of large groups of tests.

Figure 18 shows the compressive strength of about 200 specimens plotted against the percentage of summerwood as measured under a microscope. In general, the strength increased with increase in summerwood; the wide dispersion of points indicates that the relations are not well defined and, furthermore, differ considerably with the species. Shortleaf pine had the least range in summerwood but showed the greatest change in strength over that range; loblolly pine had the greatest range in summerwood and the least change in strength; longleaf pine was intermediate. When the three species were considered together, wood of average strength (3500 to

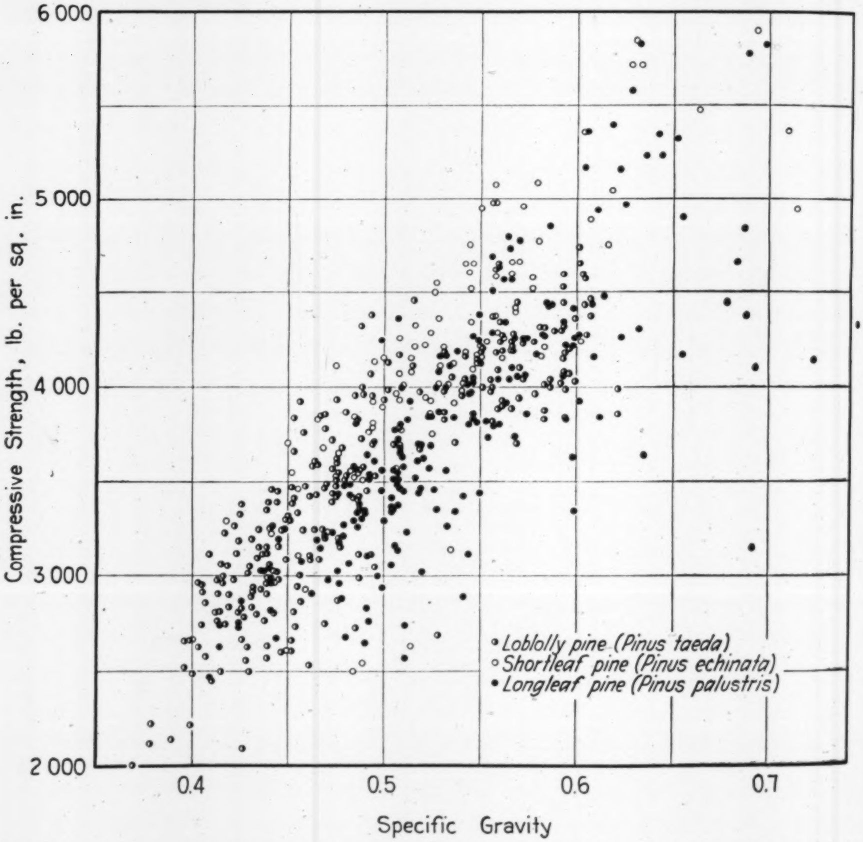

FIG. 16.—Compressive Strength Parallel to the Grain Plotted Against Specific Gravity for Three Species of Pine, Green.

Each point represents a single test.

4000 lb. per sq. in.) varied in summerwood content from 20 to 65 per cent and wood of average summerwood content (35 to 45 per cent) varied in strength from 2500 to 6000 lb. per sq. in. The average summerwood content of the specimens shown in Fig. 18 was 41 per cent for loblolly and 42 per cent for shortleaf and longleaf pine; the corresponding averages reported by Newlin and Wilson in 1917(3) were 42, 39, and 40 per cent for loblolly, longleaf and shortleaf pine, respectively. The average densities agreed to a similar degree.

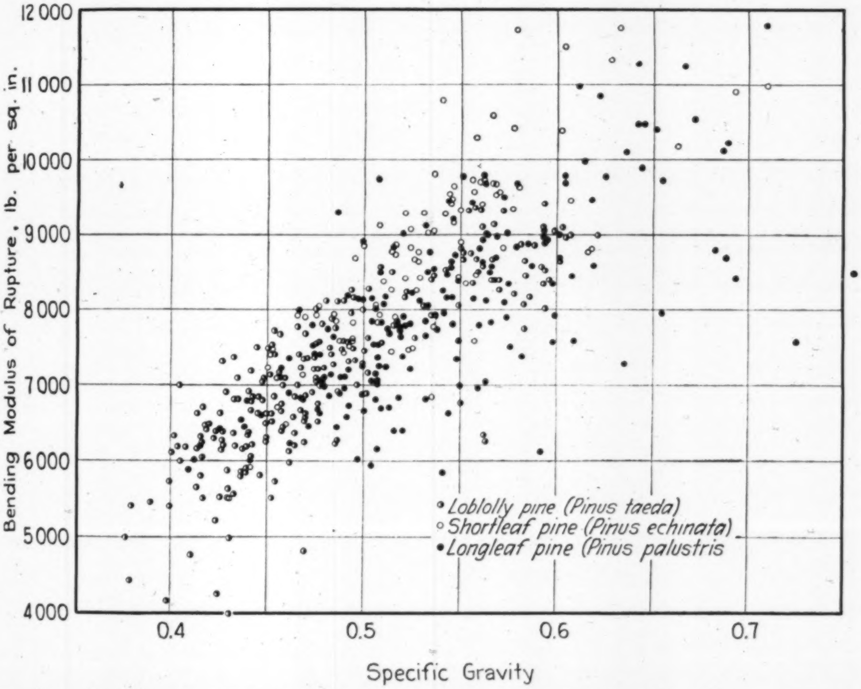

FIG. 17.—Bending Strength Plotted Against Specific Gravity, Green.
Each point represents a single test.

The modulus of elasticity increased with the density in both compression and bending but the relation was poorly defined at either very low or very high densities. Very low density was found either near the pith of rapid growth trees or near the bark of old trees; very high density was associated with high resin content or large areas of compression wood or crooked fibered material in butt logs. It was pointed out earlier that strength per unit volume varied more than weight per unit volume when wood of these various types was compared. Figure 19 shows modulus of elasticity in compression plotted against the average strength of all the specimens in one bolt; results of bending tests, similarly plotted, show almost the same degree of coordination. Each point in Fig. 19 represents from 12 to 50 tests. Modulus of elasticity, both in compression and bending, doubled while the strength increased only about 50 per cent. It should be noted that these values refer to clear, green specimens, tested under identical conditions; the increase in modulus of elasticity from green to seasoned condition is much less than the corresponding increase in strength.

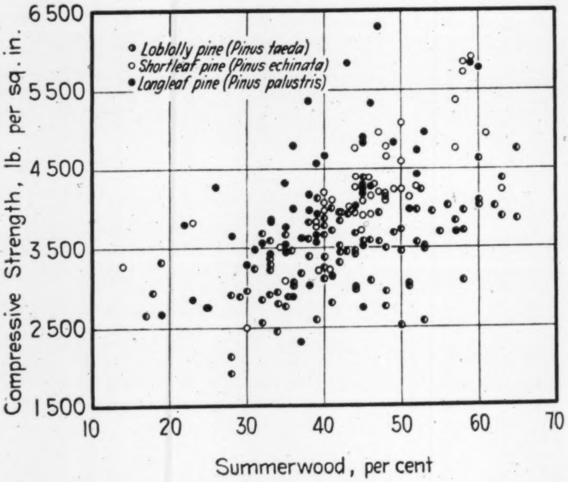

FIG. 18.—Compressive Strength Plotted Against Percentage of Summerwood (by Measurement).

Each point represents a single specimen.

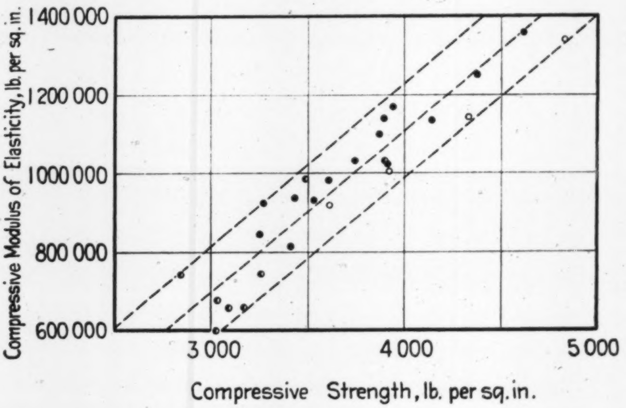

FIG. 19.—Modulus of Elasticity Plotted Against Compressive Strength.

Each plotted point represents the average of all the tests made on a single bolt or on two adjacent bolts as given in Table II.

The values reported as modulus of elasticity in compression are from 25 to 30 per cent lower than they would have been if A.S.T.M. Methods of testing had been followed, but the variations in these values are independent of differences in the two methods of testing. This is clearly indicated by the similarity of variations in compression and bending. If we consider that the stiffness of wood across the grain is only a small fraction of that along the grain and that the stiffness of a curved column is much less than that of a straight one, it is readily seen why the stiffness of crooked fibered wood or compression wood with its torquimural cells is much reduced, even if the density remains the same.

Proportional limit values showed greater and more irregular variations than any of the other mechanical properties studied. Proportional limits in compression were quite sharply defined in individual specimens and are somewhat more reliable than in bending where the deviation of the stress-strain diagram from a straight line is much more gradual and where the longer specimen allows a greater range in quality of wood. It will be noted from the bar diagrams showing individual tests that proportional limits vary in individual cases from very low values to nearly the breaking strength but the ratio of proportional limit to maximum compressive strength (or modulus of rupture), averaged by bolts and shown in the second column from the right in Tables II and III, varies relatively little for all grades of wood tested. It is worthy of note that the average ratio of proportional limit to maximum strength in compression shows little difference between green and seasoned wood and is equal to approximately 0.7. In bending, this ratio is consistently less than 0.5 for green material, but is considerably greater in seasoned material with the exception of the butt bolt of tree No. 5 which contained a large proportion of short, very crooked fibers.

These average ratios are almost identical with endurance limits in compression and bending of similar specimens under repeated but not reversed loading.[1] This strongly suggests that variations in proportional limit, not clearly reflected by changes in maximum strength or density, are due to localized areas of high stress. The variable deformation over the length of compression specimens, discussed under methods of testing, further confirms this view.

DISCUSSION

The principal argument advanced heretofore in explanation of the relations found to exist between density and strength of wood is that, since the density of wood substance itself is uniform or nearly so, the unit strength depends upon the mass per unit volume. Test material used to determine the density-strength relations was taken from "representative trees" of the species; and, further, to promote uniform comparison of species, most of the specimens were taken from a single height in the tree, namely the upper 4 ft. of the first 16-ft. log. Material from this region in the tree usually shows minimum variability, has the highest strength and elasticity in terms of its weight, and is not necessarily representative of wood near the butt or top of the tree where the greatest variations from the average are usually found. By dealing with the average strength of a cross-section, the remaining variations are minimized; in such cases the density and summerwood content are remarkably good criteria of strength.

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[1] Unpublished thesis for C. E. Degree, University of Nebraska, A. W. Brust (1931).

Since the purpose of the present study was to determine the range of variations and their causes, test material within a species was selected to include the greatest possible variations in density, growth rate, strength, and structure.

Density and summerwood content were found to become less and less accurate indices of strength as the fiber structure, age and general growth conditions became more dissimilar. The difference between weak and strong, tough and brittle, light and heavy woods has been properly ascribed to differences in the arrangement of their structural elements. The differences in mechanical properties of wood of the same species, density and moisture content must be explained on the same basis.

A review of the bar diagrams (Figs. 2, 4, 6, 8, 10, 12, and 14) shows that variations in compressive strength are accompanied by variations of the same sense and about the same relative magnitude in bending strength, stiffness in compression and bending, and somewhat less precisely by variations in the proportional limit in bending and compression. Berkley's(1) observations on the effect of certain structural variations on the compressive strength may therefore be considered also to apply to the bending strength, stiffness and proportional limit. In short, wood strongest for its weight contained only long, straight, uniform tracheids or fibers in which the elements of the fibers themselves were parallel to the axis of the fibers, and a minimum of resin ducts and ray tissue. Wood with any variation from this type of structure, whether in the form of short crooked fibers or torquimural cells[1] with resulting interstitial spaces, showed lower strength, stiffness and elasticity in terms of its weight.

Since it is obviously impractical to grade timber microscopically, other means must be used to detect structural differences. In normal, straight, uniformly grown trees showing a uniform ring structure, the amount of summerwood gives a reliable indication of strength properties for that species. Comparing species, it was found the longleaf pine containing about 30 per cent summerwood was as strong as loblolly pine with about 50 per cent summerwood if the distinction between spring and summerwood was sharp and the summerwood was dark in color. If the color distinction was poor, the relation between summerwood content and strength was almost wholly lost. Wood grown at different rates or at different ages of the tree also showed entirely different properties for the same summerwood content. Wood containing less than four rings per inch, found only in the loblolly pine, was always below average strength. Loblolly pine containing from five to twelve rings per inch was the strongest of that species whereas the same growth rate in longleaf or shortleaf pine indicated the poorest wood of those species. More than fifteen rings per inch produced the strongest wood in longleaf or shortleaf pine but in the case of loblolly pine, such slow growth occurred only in the outer few inches of old trees and was much weaker than the more rapidly grown wood formed earlier. The slowest-growth wood formed before the tree was from 80 to 100 yr. old was, in general, the strongest, but after the tree reached maturity, the strength decreased sharply with the rate of growth. No direct independent relation was found between growth rate and strength, but abrupt changes in growth rate always indicated similar abrupt changes in strength.

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[1] See p. 648.

In dense, narrow-ringed wood, the differentiation between springwood and summerwood was usually sharp and distinct in both color and thickness of cell wall. As the rings increased in width, the change from thin-walled springwood to thick-walled summerwood was more gradual and the total amount of wood substance per unit volume was less than in close-ringed wood of the same estimated summerwood content.

Rapid-growth trees often contain nearly twice as much summerwood near the butt as higher in the tree; in long timbers, summerwood content at the top gives a better index of quality than when measured at the butt. Timbers which show irregular growth rate or rings of variable width in any part of the cross-section should be viewed with suspicion regardless of the amount of summerwood present. The amount of timber marketed from second growth and cut-over stands is constantly increasing. Much of this timber shows large variations in growth rate across the section of the log. The wide-ringed wood near the pith of trees grown in the open for example (trees Nos. 1 and 2) is weak, brittle and has poor elastic properties. The wood formed later is often exceptionally strong and of fine quality generally. As a result, the *average* strength of small specimens cut from such a log may be about the same as that from a uniformly grown tree.

Large timbers cut from non-uniformly grown trees cannot be expected to react under stress in the same manner as uniform timbers of the same average strength since they are likely to be twice as strong in compression, shear and tension on one side as on the other. Unit deformation under load for specimens at the same moisture content changes relatively more than the maximum strength. It follows that a non-uniform timber does not deform as a unit; the differential deformations between the weak and strong portions cause stress distributions entirely different than those assumed in conventional design formulas. The total strength of a non-uniform timber is, therefore, greatly dependent upon the strength distribution in the piece and the manner of proportioning and loading the timber.

Of the southern pines studied, loblolly pine, frequently marketed as shortleaf pine, furnishes both the strongest and weakest wood per unit weight. Moreover, it shows the widest variation in summerwood content with the smallest corresponding change in strength, and should therefore be classified primarily on the basis of structure. The diagrams of loblolly pine trees (Figs. 3, 5, 7 and 9) show typical growth characteristics. Wood formed before the tree is about 6 in. in diameter is light, weak and brittle, but the wood formed from that time until the tree is about 100 yr. old compares favorably in strength with longleaf and shortleaf pine of the same density, although it is somewhat lacking in stiffness. Wood formed after the tree is about 100 yr. old is usually fine grained and uniform, but is light and weak and therefore unsuited for purposes where high strength is required. Because of the early age at which the loblolly pine reaches merchantable size and its ease of reproduction when protected from fire, the loblolly pine may be made a profitable source of high-grade lumber in a minimum length of time. The low commercial status of loblolly pine lumber can be largely attributed to the marketing as loblolly pine of young poles and box-heart timbers which are inferior because of rapid growth during the early age of the tree, while much of the high-grade later-formed wood reached the market as longleaf or shortleaf pine.

Longleaf pine, particularly from second-growth stands, also shows considerable variability both in structure and mechanical properties but the variations are somewhat different in type than in the wider ringed loblolly pine and their effects on the use of the wood are therefore not the same. Since the longleaf pine is characteristically a slow-growing tree, the area taken up by the inferior rings near the pith is small. The quality of pine wood depends much more upon the age of the tree than upon the distance from the pith; the slow-growing longleaf pine, therefore, produces much better poles and heart timbers than the more-rapid-growth loblolly pine. It is, however, susceptible to torquimural cells appearing as compression-wood rings.

Few specifications set a definite limit on compression wood in sawed timbers. Closer attention paid to compression wood at the mill and selective sawing to eliminate it would greatly reduce the number of timbers which might be acceptable when thoroughly green but would be rejected after seasoning because of warping or excessive shakes and checks. Examination of 1300 second-growth longleaf pine logs cut in Louisiana and Mississippi in 1934 showed that 70 per cent contained enough torquimural cells to be visually evident and 20 per cent contained compression-wood rings to the extent that parts of the logs could be rejected under specifications limiting compression wood. A group of 1100 logs consisting of shortleaf, loblolly and slash pines contained almost the same proportion of compression wood.

The greatest objection to wood containing wide rings of compression wood is based on its tendency to warp and shake due to the differential shrinkage between the concentrimural and torquimural cells. The volumetric shrinkage of the two types of structure shows relatively little difference but shrinkage along the grain was found to vary from less than 0.1 per cent in normal rings to more than 2.0 per cent in adjacent compression-wood rings. When both types of structure occur in the same timber, it usually warps badly; if the differences in structure are sufficiently great and sufficiently localized, the wood either separates between the rings or causes cross breaks in the adjacent weaker material, or both.

A review of the material and results shows that compression wood in its most obvious forms is not necessarily weaker than the adjacent wood because the loss of strength due to disturbed structure is compensated at least in part by increased density. Compression wood is nevertheless undesirable because its markedly different deformation, due either to load or to change in moisture content, causes local areas of high stress which prevent the utilization of the full strength of the remainder of the piece.

All of the variations discussed in this paper are limited to straight-grained or nearly straight-grained wood free from knots. Their importance in strength-classification purposes is therefore limited for the most part to the selection of the higher grades of timber and they are to be considered in addition to the effect of knots, cross grain and the other more obvious defects.

SUMMARY

1. The principal purpose of this investigation was to determine the magnitude and distribution of variations of certain mechanical properties, particularly those not satisfactorily explained by variations in density and summerwood content.

Maximum strength, proportional limit and stiffness in compression and bending, together with density, percentage of summerwood, rate of growth and moisture, were determined for each of about 2000 specimens of clear, straight-grained wood, systematically selected from different parts of seven southern yellow pine logs, of which four were loblolly pine, *Pinus taeda*, one was shortleaf pine, *Pinus echinata*, and two were longleaf pine, *Pinus palustris*.

2. In general, strength, elastic properties, and density decreased from the stump to the top of the log. Transversely in the tree, these properties increased from the pith to a maximum in the outer rings of young trees; in old trees, maximum values were ordinarily found between the eightieth and one hundredth rings, beyond which they again decreased toward the bark.

No consistent relation was found between strength and width of annual rings. Wood of a certain growth rate of different species or representing different ages in the tree showed widely different strength properties. Sudden changes in growth rate were accompanied by corresponding abrupt changes in strength.

3. Specimens of the same density from the same tree or from different trees from the same locality and species sometimes varied 50 per cent and more in strength and stiffness. Marked irregularities in the relations between strength, elastic properties and density were found to be caused by variations in the minute structure of the wood. Wood showing the greatest strength and elasticity (in compression and bending) per unit weight contained long, straight, concentrimural[1] tracheids or fibers and minimum resin ducts and wood rays. Specimens showing unusual weakness in terms of their density contained crooked and deformed tracheids together with numerous resin ducts and wood rays, or a high percentage of torquimural[2] tracheids, or both.

In structurally non-uniform logs, little correlation was found between summerwood content and either mechanical properties or density. Wood made up largely of irregular or distorted cells was usually found to contain a high percentage of summerwood of variable strength and density. Variation of summerwood content longitudinally in the log was found to affect strength much less than the same amount of variation in a transverse direction.

4. For fixed moisture conditions and different strengths, the moduli of elasticity in compression and bending varied proportionately more than the strengths. The relation between stiffness and strength was quite definite even in those cases of irregular fiber structure where density and summerwood content indicated strength properties least accurately.

5. Wide variations in structure over the length or cross-section of a timber cause high localized stresses due to differential shrinkage upon seasoning and to unequal deformation under load. The authors believe that much of the variation and uncertainty in fixing the elastic strength of timbers is due to such localized stresses which overload certain areas before the timber as a whole has been stressed to its limit and that serious warping and shakes are due to the same cause.

6. Extensive areas of inferior structure are readily detected visually on a cross-section by differences in color or by abrupt changes in rate of growth or summerwood content. Observation of these items is sufficient to detect serious structural defects in the clear wood of single timbers. Since the relation of strength to growth rate and summerwood content differs appreciably between the typically slow-growing longleaf and true shortleaf pines, and the more-rapid-growth loblolly and other southern pines, which in some sections are marketed indiscriminately as shortleaf pine, better methods of differentiation are still desirable.

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[1] See p. 647.

[2] See p. 648.

Acknowledgments:

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METHODS OF TESTING WOOD TREATED WITH FIRE RETARDANTS[1]

By C. R. Brown[2]

Synopsis

The studies described in this paper were made to compare the fire-tube test and the flame-penetration test as procedures for indicating some of the properties relating to the tendency of wood to support combustion and spread fire and the effect of treatments upon these properties. Several factors affecting the results of each test were studied, using specimens of untreated and treated wood varying in species, source, and treatment.

It was found that the results of both tests are not affected to any appreciable extent by moisture contents of specimens when within the normal range. The loss in weight in grams and certain other results are directly related to density, but otherwise density is not important. Wood species, source, nature and concentration of treatment were found to be important factors in both tests, and thickness of specimen is also a factor in the flame-penetration test. Treatments which reduce combustibility do not necessarily retard flame penetration.

It is concluded that the fire-tube test is a measure of the tendency of wood to spread fire and differentiates between untreated, poorly-treated, and well-treated wood. The flame-penetration test also gives some information on the same point but does not give as sharp distinctions. It has the advantage that no special equipment is required, shorter samples can be used for specimens, and fewer specimens are needed to obtain average results representative of a given lot of material.

Introduction

From time to time, as general interest in the use of fire-retardant treated wood has been revived, various attempts **(1-38)**[3] have been made to establish adequate methods for determining the effect of different treatments upon those properties involved in the fire hazard and fire resistance of wood in its many uses. While many of these tests have been deemed unsatisfactory, some appear to be capable of development into useful procedures, and a few have been studied intensively with this in view.

Several writers **(1,11,24,28,29,35)** have indicated that any testing procedure should give definite information in numerical terms on at least one of the following points:

1. Ease of ignition of the material.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Assistant Physicist, Fire Resistance Section, National Bureau of Standards, Washington, D. C.

[3] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 693.

2. Its tendency to support combustion and spread fire, giving off flammable gases and developing heat, that is, its contribution to fire.
3. The tendency of the material to continue to glow.
4. Its ability to retard the transmission of heat developed by the fire.
5. Its ability to resist penetration of fire through the material.
6. Loss of strength and other structural properties when exposed to fire.

Two procedures, the fire-tube test and the flame-penetration test, have been studied at the National Bureau of Standards primarily for the purpose of obtaining data which may be of value in preparing specifications for testing wood treated with fire retardants. Accordingly, several factors which might affect the results of each test have been considered, using untreated and treated wood of different species and from various sources. Attempts have also been made to compare the results by one test with those obtained by the other on the same lots of material. This paper presents the principal results of these studies with particular reference to the extent to which these methods indicate the tendency of wood to support combustion and spread fire.

FIRE-TUBE TESTS

Method:

(*a*) *Procedure.*—Since the fire tube as devised by the Forest Products Laboratory, Department of Agriculture, has been described elsewhere(16,24,28,29), only the essentials will be given here. The apparatus consists of a sheet-metal tube suspended from one arm of a balance beam. A $\frac{3}{8}$ by $\frac{3}{4}$ by 40-in. specimen is hung centrally in the tube and a thermocouple is located just above the upper end of the specimen. A pointer attached to the center of the beam serves to indicate loss in weight in percentage on an adjustable scale. After preliminary adjustments, a bunsen burner is applied 1 in. below the lower end of the specimen for 4 min. Temperature and weight loss readings are taken at frequent intervals until all flaming of the specimen has ceased.

Except for minor changes, the fire-tube tests reported in this paper were made in the manner specified by the Forest Products Laboratory. Although the flame was adjusted to the specified length of 11 in. and to give a maximum indicated temperature in the flame of 1000 C., it was found that the temperature at the top of the tube seldom approached the 180 C. also specified. After about 140 tests had been made with the 11-in. flame it was decided that it would be more desirable to adjust the flame to give a temperature of 180 ± 5 C. at the top of the tube, and this has been done in subsequent tests. This flame was only slightly longer than that for previous tests and the change apparently had no significant effect on the results for untreated wood, although there is some evidence of a slight increase in maximum temperatures for well-treated specimens.

The Forest Products Laboratory usually reports fire-tube results in terms of the final loss in weight in percentage[1] and the maximum temperature observed during the test. To these we have added the area under the temperature curve measured above the 180 C. line, the weight loss in grams, and the maximum rate of loss occurring in any one minute in grams per minute, for the purpose of obtaining additional information.

(*b*) *Possible Modifications.*—Most of the details of procedure which might influence the results have already been studied thoroughly by the Forest Products Laboratory(16,24,28) and accordingly the method has been standardized in most respects. In our study some attention was given to the possibility of reducing the time of exposure to the burner flame. While a 1-min. application was found to be sufficient for untreated woods, at least 3 min. was necessary for well-treated specimens, and later experience showed that a 4-min. exposure was sometimes required to indicate the presence of poorly-treated material. Consequently, a change in the time of burner application is not recommended.

TABLE I.—TYPICAL VARIATIONS IN FIRE-TUBE RESULTS FOR INDIVIDUAL SPECIMENS FROM SAME LOT.

Result	Minimum and Maximum Values					
	Untreated Shortleaf Pine		Poorly-Treated Longleaf Pine		Well-Treated White Pine	
	Min.	Max.	Min.	Max.	Min.	Max.
Weight loss, per cent	86	87	25	55	17	21
Weight loss, g	92.4	101.5	20.2	47.0	13.6	17.3
Maximum temperature, deg. Cent	763	842	185	588	162	185
Area under temperature curve, deg. Cent.-min	1622	1776	6	382	....	11
Maximum rate of loss, g. per min	34.7	38.5	5.6	17.1	4.8	5.8

Also, the use of a finer wire for the thermocouple seemed desirable in order to reduce lag and conduction away from the junction. The present 14-gage wire is unnecessarily heavy for the service required. While several tests with treated wood in which 23 and 26-gage wires were used confirmed this assumption, the 14-gage thermocouple was retained in the remaining tests to conform to the specified procedure.

(*c*) *Reproducibility of Results.*—Table I indicates typical variations for untreated, poorly-treated, and well-treated material, for lots of similar specimens. The variations in the weight loss in percentage are small for all untreated lots of material. The large range in results for poorly-treated specimens is probably accounted for in part by the variability of the material itself. Admitting the latter factor as well as uncontrollable differences in specimens, the reproducibility of results on truly identical specimens would be at least as good as, and probably better than, that shown in Table I.

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[1] The loss in weight reported herein is that recorded 2 min. after flaming ceased.

TABLE II.—AVERAGE RESULTS FOR VARIOUS WOOD SPECIES, SOURCES, AND TREATMENTS.

Specimen Data					Loss in Weight		Maximum Temperature, deg. Cent.	Area Under Temperature Curve–deg. Cent.–min.	Maximum Rate of Loss, g. per min.
Species	Source and Lot	Number of Specimens	Moisture, per cent	Density, g. per cu. cm.	per cent	grams			
			UNTREATED SPECIMENS						
White pine	E-1	4	5.8	0.38	81	56.8	732	1468	24.7
Western yellow pine	A-1	3	6.2	0.42	87	67.0	729	1375	28.5
Longleaf pine	A-2	6	8.2	0.60	83	91.5	733	1723	30.6
	B-1	2	8.2	0.52	83	78.5	774	1653	29.4
	B-3	6	8.6	0.62	83	95.4	731	1697	31.8
	B-6	4	10.8	0.67	83	103.2	718	1914	32.8
Shortleaf pine	A-3	5	7.2	0.60	87	95.3	799	1692	36.7
	A-4	5	10.5	0.61	84	95.3	756	1681	31.6
Douglas fir	J-1	5	10.5	0.54	82	76.1	736	1830	23.6
Oak	A-5	3	5.1	0.69	82	104.0	796	1703	40.4
	C-3	4	5.1	0.55	82	83.0	662	1413	32.5
Birch	C-6	2	6.8	0.75	86	118.1	693	1751	39.4
	E-3	4	5.6	0.66	82	99.3	728	1687	35.5
Chestnut	E-5	4	6.7	0.50	75	69.6	702	1433	27.1
Black walnut	C-12	2	7.0	0.58	84	90.8	792	1742	31.7
	E-8	4	6.0	0.64	82	97.6	733	1620	34.7
	E-10	4	5.2	0.58	79	85.2	785	1644	33.8
Black cherry	E-12	4	6.7	0.57	76	80.6	722	1504	31.1
			TREATED SPECIMENS						
White pine	E-2	4	5.3	0.44	19	15.8	172	[a]	5.3
	G-1	6	12.8	0.46	19	15.7	138	....	4.8
	G-2	5	12.6	0.44	17	14.0	157	[a]	4.3
Western yellow pine	D-1	3	8.4	0.44	21	17.1	167	....	6.0
Longleaf pine	B-2	4	7.2	0.58	14	14.9	132	....	5.2
	B-4	6	7.4	0.69	19	23.4	183	[a]	7.5
	B-5	6	7.4	0.68	18	22.8	174	[a]	7.2
	B-7	4	9.5	0.64	18	21.5	177	[a]	7.0
	B-8	4	9.2	0.64	18	21.4	180	[a]	6.6
	F-1	5	6.5	0.42	35	29.0	321	152	11.0
	F-2	5	8.6	0.45	35	29.5	267	[a]	9.8
White cedar	C-1	6	5.2	0.57	12	12.8	136	....	3.7
	C-2	6	4.5	0.60	14	14.9	165	....	4.4
Douglas fir	D-2	10	15.4	0.56	21	21.0	178	[a]	6.4
Oak	C-4	6	4.4	0.66	13	13.6	136	....	4.9
	D-3	3	5.4	0.83	18	26.9	172	[a]	8.1
Birch	C-5	3	....	0.78	14	20.6	163	....	6.2
	C-7	6	6.6	0.78	15	21.4	154	....	6.3
	C-8	5	5.5	0.69	27	31.7	234	[a]	13.2
	C-9	5	9.0	0.68	27	34.1	214	35	12.6
	C-10	5	10.8	0.63	25	28.7	184	[a]	9.9
	D-4	3	6.4	0.72	25	32.8	228	[a]	11.5
	E-4	4	6.0	0.76	22	31.2	204	25	10.5
Chestnut	C-11	6	4.2	0.63	11	12.7	123	....	4.0
	D-5	3	6.7	0.47	18	15.4	163	....	4.6
	E-6	3	5.9	0.58	18	22.5	182	[a]	6.0
	E-7	4	5.9	0.58	33	33.8	322	[a]	11.3
Black walnut	C-13	6	....	0.75	15	20.5	150	....	6.4
	D-6	3	6.2	0.69	14	18.2	141	....	6.0
	D-7	6	7.3	0.65	18	22.1	142	....	6.6
	E-9	4	6.0	0.72	46	60.6	418	758	17.3
	E-11	4	6.1	0.62	47	53.3	426	638	17.8
Black cherry	E-13	4	7.0	0.62	48	55.4	398	549	15.2
Maple	D-8	3	6.8	0.65	22	26.4	179	[a]	7.6
	D-9	5	9.0	0.72	25	36.2	181	[a]	10.3

[a] Some specimens exceeded 180 C., but not all.

While few data are available for direct comparison, our studies as well as those made by the Forest Products Laboratory indicate that the average fire-tube results for given lots of untreated and well-treated material are reproducible to within about 5 per cent. Much depends upon the method of selecting specimens, and although we have regarded five or more, if properly chosen, as fairly representative of a given lot, the Forest Products Laboratory (24) suggests four specimens from each of four boards.

Results:

Table II is a summary of the average results of all fire-tube tests on different lots of wood from several sources (indicated by letters) and representative of various species and treatments. The nature and concentration of treatment are not given as these could not be ascertained for most of the commercial treatments. Densities are based on the weight of the specimen (including moisture) and volume at time of test. Where the temperatures for some specimens in a particular lot did not exceed 180 C. (as indicated by a footnote), a proper average for the area under the temperature curve cannot be determined.

TABLE III.—RELATIVE EFFECTIVENESS OF TREATMENT.

Material			Decrease in Maximum Temperature, deg. Cent.	Ratio of Result for Treated Wood to Result for Untreated Wood		
				Loss in Weight		Maximum Rate of Loss
Species	Un-treated Lot	Treated Lot		per cent	grams	
White pine..................	E–1	E–2	560	0.23	0.28	0.21
Longleaf pine...............	B–1	B–2	642	0.17	0.19	0.18
	B–3	B–4, 5	553	0.22	0.24	0.23
	B–6	B–7, 8	540	0.22	0.21	0.21
Oak.........................	C–3	C–4	526	0.16	0.16	0.15
Birch.......................	C–6	C–7	539	0.17	0.18	0.16
	E–3	E–4	524	0.27	0.31	0.30
Chestnut....................	E–5	E–6	520	0.24	0.32	0.22
Black walnut................	C–12	C–13	642	0.18	0.23	0.20
	E–8	E–9	315	0.56	0.62	0.50
	E–10	E–11	359	0.59	0.63	0.53
Black cherry................	E–12	E–13	324	0.63	0.69	0.49

(*a*) *Species and Source.*—Comparing the results for the untreated wood, it is seen that the weight loss in percentage is about the same for all lots, but the rate of weight loss is somewhat different for different species. It is also evident that hardwoods as a class do not necessarily give lower values for any of the results than softwoods. For treated woods, the importance of species is difficult to evaluate, since the nature and concentration of treatment are important in the latter case.

(*b*) *Treatment.*—Table II also permits a direct comparison of different lots of treated wood and indicates the range in fire-tube results to be expected for the several species treated by various methods. In order to compare the effectiveness of the different treatments, the ratio of each of the weight-loss results for treated wood to the comparable figure for untreated wood from the same source has been given in Table III. This table also includes the average reductions in maximum temperatures. Presented in this way, the data indicate that certain treatments appear to be more effective than others, and the weight loss and rate of loss for at least some species can be reduced from 70 to 85 per cent by proper treatment.

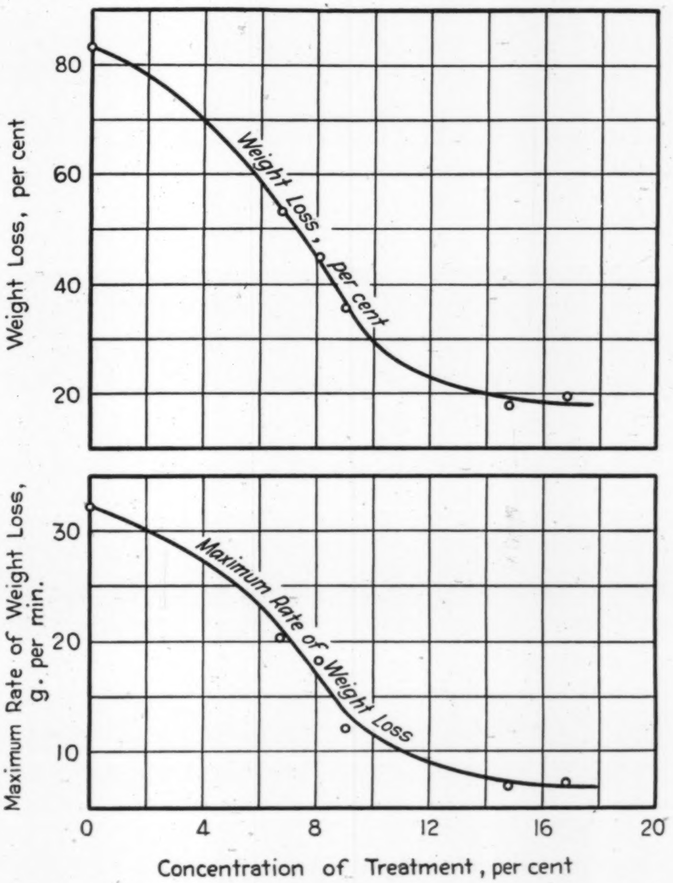

FIG. 1.—Relations Between Weight Loss in Percentage and Maximum Rate of Weight Loss, and Concentration of Treatment for Longleaf Pine Specimens Treated with Monoammonium Phosphate.

The effect of concentration of treatment has been adequately studied by Truax and others(16,24,28,29,39,40,41,42) and while not extensively covered in the present tests, such data as we have obtained agree with their results.

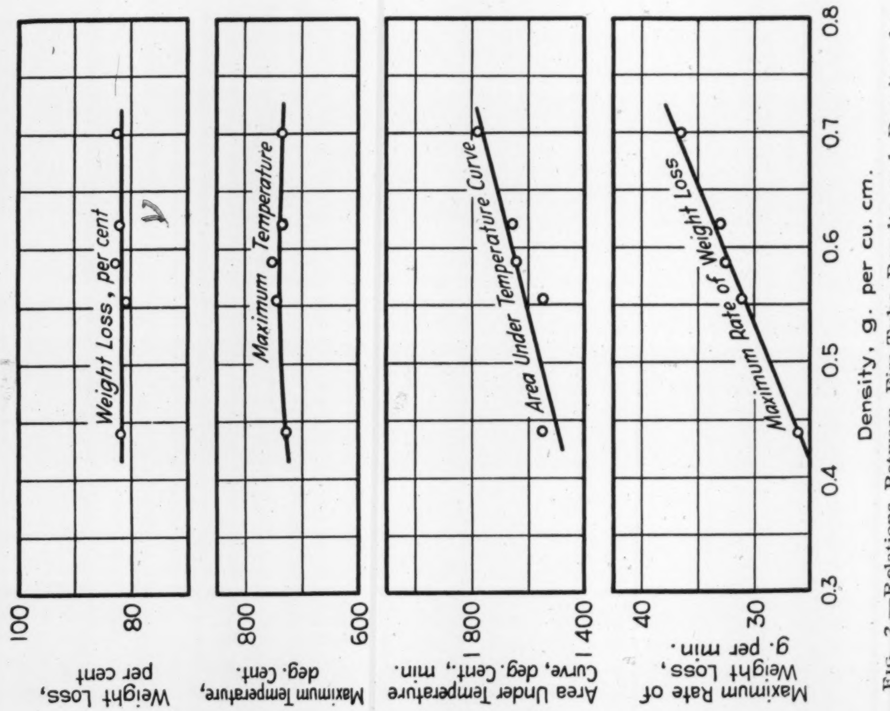

FIG. 2.—Relations Between Fire-Tube Results and Density, for Untreated Specimens.

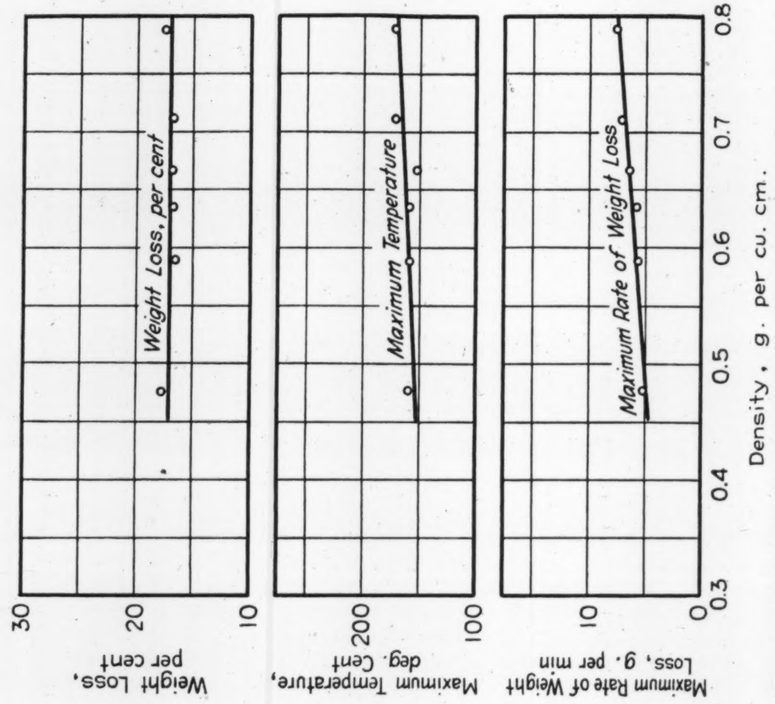

FIG. 3.—Relations Between Fire-Tube Results and Density, for Well-Treated Specimens (Less than 25 per cent Loss in Weight).

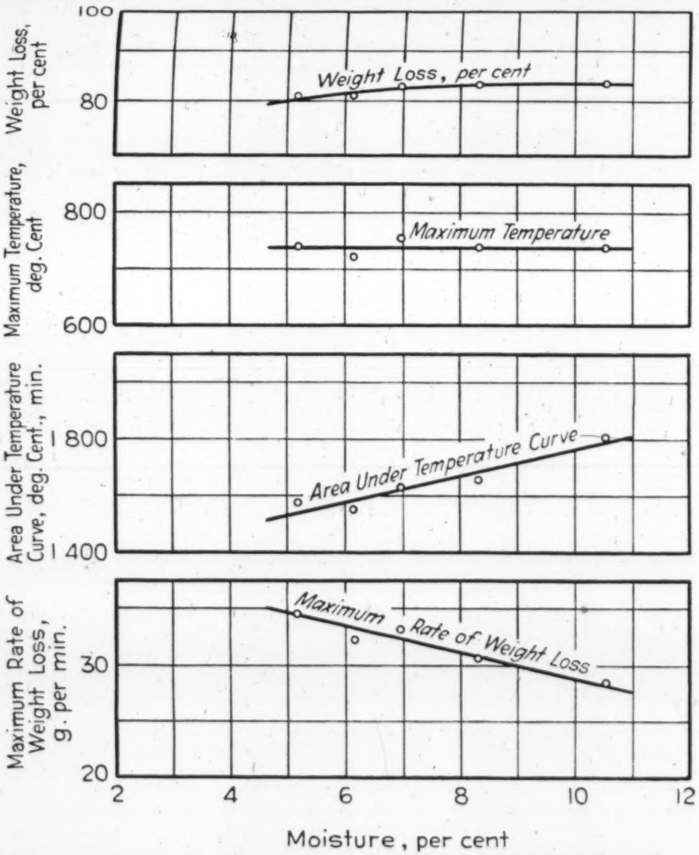

FIG. 4.—Relations Between Fire-Tube Results and Moisture, for Untreated Specimens.

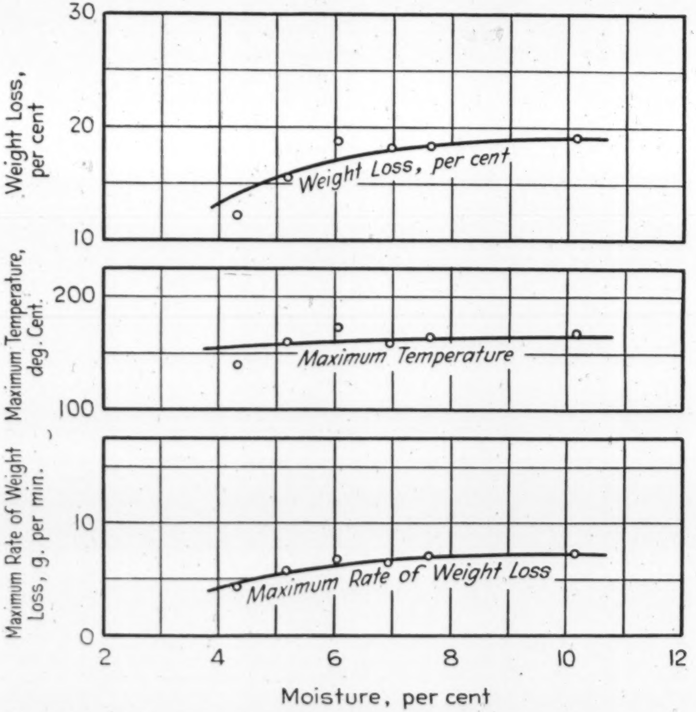

FIG. 5.—Relations Between Fire-Tube Results and Moisture, for Well-Treated Specimens (Less than 25 per cent Loss in Weight).

Figure 1 shows the average relations between two of the fire-tube test results and concentration of treatment[1] for longleaf pine treated with monoammonium phosphate. Similar relations hold for other test results and it is evident that little is gained by increasing the treatment beyond a certain concentration.

(*c*) *Density.*—While appreciating that density is related in a general way to species, the following study of its effect has been made without differentiation between species. Each of two classes of individual results, one including all untreated specimens, and the other those treated specimens for which the loss in weight did not exceed 25 per cent, were averaged in groups arranged according to density. Each group was large enough to include specimens from more than one source and all groups in each class had the same average moisture content. As indicated in Figs. 2 and 3, the weight loss in percentage is independent of density for both untreated and well-treated material. Consequently, since the size of specimen is constant, the weight loss in grams (not shown on plots) is directly proportional to density. The maximum temperature is apparently only slightly affected, but the area under the temperature curve and the maximum rate of loss both increase with density.

TABLE IV.—AVERAGE RESULTS OF SPECIAL MOISTURE STUDY.

Specimen Data				Loss in Weight		Maximum Temperature, deg. Cent.	Area Under Temperature Curve, deg. Cent.-min.	Maximum Rate of Loss, g. per min.
Species	Type	Moisture, per cent	Density, g. per cu. cm.	per cent	grams			
SPECIMENS CONDITIONED AT 80 F. AND 30 PER CENT HUMIDITY								
Shortleaf pine	untreated	7.2	0.60	87	95.3	799	1692	36.7
Longleaf pine	treated	6.5	0.42	35	29.0	321	152	11.0
Birch	treated	5.5	0.69	27	31.7	234	[a]	13.2
SPECIMENS CONDITIONED AT 70 F. AND 65 PER CENT HUMIDITY								
Shortleaf pine	untreated	10.5	0.61	84	95.3	756	1681	31.6
Longleaf pine	treated	8.6	0.45	35	29.5	267	[a]	9.8
Birch	treated	9.0	0.68	27	34.1	214	35	12.6
SPECIMENS CONDITIONED AT 70 F. AND 100 PER CENT HUMIDITY								
Shortleaf pine	untreated	95.1	1.10	18	35.4	149	....	9.3
Longleaf pine	treated	139.3	0.90	15	29.0	129	....	7.9
Birch	treated	73.4	1.10	15	31.1	137	....	9.3

[a] Some specimens exceeded 180 C., but not all.

(*d*) *Moisture.*—The averages for a similar study of the effect of moisture, in which the groups varied in moisture content but had the same average density, are plotted in Figs. 4 and 5. The curves for the weight loss in grams have been omitted since they are identical in shape with those for weight loss in percentage, inasmuch as the average density is constant. It is seen that weight loss in percentage is affected more by the moisture content in the case of treated specimens than in the case of untreated specimens, apparently because moisture makes up a greater proportion of the total loss for treated wood than for untreated. The maximum temperature seems to be little affected in the range of moisture studied. The fact that the maximum rate of loss decreases with increasing moisture content of untreated wood may explain why the area under the temperature curve increases, since the specimen burns longer and maintains high temperatures for a greater period.

---

[1] Weight of dry salt expressed as percentage of weight of air-dry wood.

In a second study of the effect of moisture, matched lots of five specimens each were tested after storage under the three different conditions stated in Table IV, which gives average results for the tests. Although a relative humidity slightly less than 100 per cent was intended for the last condition, condensation occurred to such an extent that the specimens tended to become waterlogged. The results do show, however, that while abnormal moisture has a decided fire-retardant effect, particularly in the case of untreated woods, moisture content in the range from about 6 to 12 per cent has little or no effect.

(*e*) *Veneer Specimens.*—About 30 tests were made on built-up specimens, constructed by nailing together two or more plies of veneer to obtain the required thickness of $\frac{3}{8}$ in. On the whole the results are quite comparable with those for solid specimens except that the maximum rate of weight loss seems to be somewhat higher for the veneer specimens, probably because the plies tend to separate while burning.

Summary:

From the results of the fire-tube tests, it may be concluded that:

1. The only changes in procedure to be recommended are the use of finer wires for the thermocouple, and the adjustment of the flame primarily to give a temperature of 180 C. at the top of the tube, but otherwise conforming as nearly as possible to the 11-in. flame specified.

2. The reproducibility of results in the fire tube appears to be satisfactory, considering the variability of wood itself. At least five, and possibly ten, specimens are necessary to obtain representative average results for a given lot of material and it is important that the specimens be properly selected.

3. Moisture in the range from about 6 to 12 per cent has little or no effect on most of the test results, hence a narrow range in moisture content of specimens at the time of tests is not necessary.

4. The fire-tube test distinguishes between untreated and well-treated wood, and also indicates the degree of treatment to some extent. For well-treated specimens the weight loss ranges from 10 to 25 per cent and

the maximum indicated temperature does not exceed 250 C. As the effectiveness of treatment becomes less, a greater amount of wood is consumed and much higher temperatures are often observed, until for untreated wood the weight loss ranges from 75 to 87 per cent and the maximum temperatures reach approximately 750 C.

5. The weight loss in percentage is not influenced by density, nor does it vary much with species, or with moisture content within the above range.

6. The maximum temperature is affected little by density or moisture within the normal range, but is variable from piece to piece. Although it might be expected to bear a relation to the rate of heat development, as measured in this test it is evidently affected by conditions inherent in the procedure to such an extent that it is not a reliable indication.

7. As an indication of the rate of heat development the maximum rate of weight loss shows a distinction between different species, at least for untreated wood.

8. The weight loss in grams seems to be a logical measure of the total heat developed, and since it can be determined more readily, it is preferable to the area under the temperature curve, which has been suggested (28) for this purpose.

FLAME-PENETRATION TESTS

A form of the flame-penetration test was first used at the National Bureau of Standards to indicate the penetration of fire through various wallboards and combustible insulation boards and the spread of fire over the surface after flame penetration. As a rule the tests were made in comparison with those on some accepted material. In applying the test to treated wood, the procedure has been developed with the hope of obtaining information on the tendency of wood to spread fire as well as its ability to resist flame penetration.

Method:

(*a*) *Procedure.*—As the flame-penetration test is now applied at the Bureau of Standards, the specimen is supported in a horizontal position with its center 3 in. above the tip of a $\frac{3}{8}$-in. Tirrill burner. The rate of gas flow is first regulated so that the burner produces about 3000 B.t.u. per hr. and the burner flame is then adjusted by starting with a tall inner cone and increasing the air supply until the inner cone has just reached a minimum. With the gas used in this laboratory, a flame having an outer cone of about 7 in. and an inner cone of nearly 2 in. is obtained by the above procedure. A bare thermocouple is located at the center of the top surface of the specimen. Observations are made of the time for light to come through any crack, time of flame penetration, time of ignition on the top surface, and the duration of flaming. The specimen is weighed before and after test. If ignition does not occur within 5 min. after flame penetration, the burner is then removed. The normal size of specimen is 10 in. square but any convenient size larger than 6 in. square can be used.

In addition to the time of flame penetration and other time data, the results include the weight loss in grams, the average rate of weight loss, and the nominal weight loss in percentage. The latter is the actual weight loss in percentage multiplied by the ratio of the area of the specimen to that of a specimen 10 in. square, this adjustment being made in order to place this figure on the same basis for all specimens of the same thickness. The rate of weight loss is found by dividing the total loss in weight by the time of burner application plus the duration of flaming after removal of the burner. The moisture content was determined by drying small pieces of the same board at 105 C. for about 48 hr., which was found to be sufficient for specimens weighing 100 g. or less.

(*b*) *Development.*—Part of the tests reported herein were made according to procedures differing somewhat from that described above, which has been designated as *E* in the tables. In the earliest procedures, *A*, *B*, and *C*, a bunsen burner was used with the heights of the inner and outer cone adjusted to about $2\frac{1}{2}$ and 5 in., respectively. The center of the specimen was located $3\frac{3}{4}$ in. above the tip of the burner. The burner flame was applied until the upper surface ignited, following flame penetration, but not to exceed 90 min. In procedure *C* the gas pressure was measured and kept constant during the test instead of the height of the inner cone as in previous tests. The corresponding rate of heat production was about 1900 B.t.u. per hr. Also in procedure *A* the specimen was usually 6 in. square and the edges were protected by an asbestos-cloth guard. No protection is used at present, any tendency for flame to come up over the edges being restrained by applications of a damp cloth on the edges during the first few minutes of a test.

The chief objections to these earlier methods were that with this exposure flame penetration did not always occur within 90 min., or if it did occur but without ignition of the specimen on the upper surface, no provision was made for burner removal within a reasonable time after flame penetration. Before changing the procedure again, however, a special study was made with a Tirrill and a Fisher (Meker type) burner at various gas pressures, using matched specimens of treated birch. It was found that for the same gas pressures up to about 5 in. of water, flame penetration occurs sooner when the Tirrill burner is used, but the weight loss is greater for the Fisher burner. The Tirrill burner was selected as being more suitable for flame-penetration purposes, and as little was gained by increasing the flame height above 7 in., the latter flame height was chosen for procedure *D*. This was nearly the same as the present method *E*, except that

TABLE V.—VARIATIONS IN FLAME-PENETRATION RESULTS FOR INDIVIDUAL SPECIMENS FROM SAME LOT.

All ¾-in. specimens, tested by procedure *D*.

Result	Minimum and Maximum Values			
	Untreated Shortleaf Pine		Treated Oak	
	Minimum	Maximum	Minimum	Maximum
Time to reach 100 C., min.	9.9	11.5	11.9	12.3
Flame penetration or ignition, min.	14.3	19.9	34.0	38.5
Loss in weight, g.	174	389	73	87
Nominal loss in weight, per cent	28	46	9	10
Rate of loss in weight, g. per min.	6.9	9.9	1.6	2.1

TABLE VI.—RESULTS FOR ¾-IN. SPECIMENS OF VARIOUS SPECIES, SOURCES, AND TREATMENTS.

Specimen Data						Time to Reach 100 C., min.	Time to Reach 200 C., min.	Light Seen Through, min.	Flame Penetration or Ignition, min.	Net Duration of Flaming, min.	Loss in Weight		Rate of Loss, g. per min.
Species	Source and Lot	Type	Number	Moisture, per cent	Density, g. per cu. cm.						Actual, g.	Nominal, per cent	
RESULTS BY PROCEDURE *A*													
Western yellow pine	A-1	U	1	6.4	0.50	10.5	22.1	30.2	31.9	7.1	110.5	19.9	2.83
Shortleaf pine	A-6	U	1	7.8	0.56	9.1	19.4	23.8	25.1	16.7	209.3	32.8	5.01
RESULTS BY PROCEDURE *B*													
White pine	D-10	T	1	2.1	0.45	13.9	20.9	22.1	24.1	none[a]	50.9	9.1	0.57
White cedar	C-1	T	2	3.8	0.55	18.4	[b]	[b]	[b]	....	39.8	5.9	0.44
	C-2	T	3	3.9	0.55	17.8	[b]	[b]	[b]	....	48.6	7.2	0.54
Oak	C-3	U	1	3.2	0.56	9.9	37.2	55.5	61.5	6.0	126.0	18.4	1.86
	C-4	T	2	2.5	0.66	16.0	70.5	51.3	[b]	....	55.2	6.9	0.62
Birch	C-6	U	1	4.5	0.71	11.2	22.7	35.6	39.9	8.2	139.0	15.9	2.89
	C-7	T	3	4.2	0.72	14.3	25.0	26.1	31.4	none[a]	65.9	7.5	0.73
Chestnut	C-14	U	1	4.6	0.41	11.1	40.4	35.7	43.4	5.1	78.5	15.4	1.56
	C-11	T	2	3.8	0.58	18.2	78.0	68.4	[b]	....	57.6	8.2	0.64
Yellow poplar	D-11	T	1	6.5	0.47	16.9	37.5	39.2	45.4	none[a]	60.3	10.5	0.67
RESULTS BY PROCEDURE *C*													
Black walnut	C-12	U	1	4.5	0.52	11.9	31.1	43.9	49.8	4.8	90.2	14.3	1.64
	C-13	T	3	3.7	0.61	15.7	[b]	[b]	[b]	....	47.8	6.4	0.53
RESULTS BY PROCEDURE *D*													
Sugar pine	H-1	T	1	7.1	0.53	14.3	25.1	25.6	28.5	0.0	64.9	10.0	1.94
	H-2	T	2	12.6	0.52	14.8	23.8	22.4	28.0	0.0	62.4	9.7	2.00
	I-1	T	1	3.9	0.47	13.8	23.6	23.2	27.4	0.3	63.1	10.8	1.93
	I-2	T	1	8.4	0.49	16.0	37.4	35.1	43.8	0.4	67.4	11.2	1.37
Longleaf pine	A-2	U	2	6.4	0.66	10.3	14.8	16.4	17.2	15.9	223.2	27.2	6.76
	A-2	U	2	9.8	0.68	12.2	16.4	17.2	18.0	13.0	185.8	22.6	5.98
Shortleaf pine	A-7	U	1	8.3	0.67	13.0	21.2	23.2	23.9	17.1	313.6	38.2	7.65
	A-3	U	3	6.0	0.58	10.6	15.3	17.1	18.0	13.8	258.5	35.1	7.89
	A-4	U	3	11.0	0.59	12.4	19.0	22.7	26.3	13.2	231.9	30.0	5.49
Douglas fir	A-8	U	1	8.6	0.50	17.1	35.0	37.6	43.0	4.6	97.1	15.8	2.04
Oak	C-15	T	3	4.4	0.69	12.1	24.9	27.9	36.0	0.5	81.7	9.5	1.96
	C-16	T	5	8.6	0.71	14.1	32.6	35.4	52.6	0.3	91.2	10.5	1.65
Yellow poplar	F-3	T	1	5.5	0.47	10.0	14.1	14.2	15.2	0.5	71.4	12.2	3.45
	F-4	T	2	11.9	0.50	13.0	18.4	18.4	20.3	0.2	68.8	11.4	2.69

[a] Burner flame applied 90 min., no ignition.

[b] Not obtained in 90 min.

for the latter a rate of gas flow corresponding to about 3000 B.t.u. per hr. is maintained during the test, but the flame height is still about 7 in. The test is always continued until flame penetration occurs, but not longer than 5 min. after this event.

(*c*) *Reproducibility*.—Table V indicates the variations in flame-penetration results for lots of three specimens each. Although from the same lot, the untreated pine specimens were visibly different in pitch content, while the treated oak specimens were very well matched. Nevertheless, our experience has been that results with untreated wood are usually more variable than those with treated wood.

TABLE VII.—RELATIVE EFFECTIVENESS OF TREATMENT.

Species	Untreated Lot[a]	Treated Lot[a]	Thickness, in.	Increase (+) or Decrease (−)		Ratio of Result for Treated Wood to Result for Untreated Wood		
				Time to Reach 100 C., min.	Flame Penetration, min.	Loss in Weight		Rate of Loss in Weight
						Actual	Nominal	
RESULTS BY PROCEDURE *B*								
Oak	C-3	C-4	0.50	+1.3	−0.6	0.54	0.40	0.16
	C-3	C-4	0.75	+6.1	(+28.5)[a]	0.44	0.38	0.33
Birch	C-6	C-7	0.50	+2.2	+6.3	0.21	0.19	0.09
	C-6	C-7	0.75	+3.1	−6.8	0.47	0.47	0.25
Chestnut	C-14	C-11	0.50	+2.6	−1.7	1.22	0.74	1.22[b]
	C-14	C-11	0.75	+7.1	(+46.6)[a]	0.73	0.53	0.41
RESULTS BY PROCEDURE *C*								
Black walnut	C-12	C-13	0.50	+2.4	+18.2	0.44	0.37	0.17
	C-12	C-13	0.75	+3.8	(+40.2)[a]	0.53	0.45	0.32
RESULTS BY PROCEDURE *E*								
White pine	E-1	E-2	0.84	+4.0	+38.5	0.83	0.72	0.40
Birch	E-3	E-4	0.88	+4.3	+0.5	0.58	0.49	0.58
Chestnut	E-5	E-6	1.06	+3.6	+36.0	1.13	0.81	0.58
..	E-20	E-7	1.76	+58.5	+107.4	1.12	0.86	0.43
Black walnut	E-14a	E-15a	0.06	......	−0.05	0.06	0.06	0.03
	E-14b	E-15b	0.12	......	+1.1	0.14	0.10	0.12
	E-8	E-9	0.88	+0.1	+25.0	0.65	0.58	0.53
	E-10	E-11	1.41	+42.8	+125.1	1.22	0.93	0.51
Yellow poplar	E-16a	E-17a	0.06	......	+0.15	0.08	0.06	0.04
	E-16b	E-17b	0.12	......	+0.65	0.15	0.08	0.07
Black cherry	E-18a	E-19a	0.06	......	+0.08	0.09	0.05	0.06
	E-18b	E-19b	0.12	......	+0.2	0.17	0.14	0.13
	E-12	E-13	0.88	+4.1	+1.3	0.83	0.70	0.78

[a] a = Exact amount indeterminate, but at least the amount shown as flame penetration in the treated specimen had not occurred at 90 min. b = Untreated specimen failed to ignite upon flame penetration.

Regarding the number of specimens required in order that the average results may be representative of a lot of material, it was found in the study with built-up specimens (not here reported) that average results for two separate sets of three specimens each from the same lot were in good agreement. It is believed, therefore, that from three to five specimens of the same thickness and selected from different portions of several boards would be sufficient for the purpose.

Results:

The results for all $\frac{3}{4}$-in. specimens of normal moisture content, or about one-third of the total tested, are listed in Table VI. All procedures are represented except *E*, for which there were no $\frac{3}{4}$-in. specimens. The effect of the use of a more intense burner flame can be observed by comparing results by procedures *A*, *B*, and *C* with those by procedure *D*.

(*a*) *Species and Treatment.*—It is evident that species influence the time of flame penetration as well as the weight-loss results. For treated specimens, it is probably more important than the nature of treatment. The data in Table VI show that treated specimens are usually but not necessarily more resistant to flame penetration than untreated specimens. Also, the highest values of weight loss in grams or percentage for treated wood approach the lowest values of the same results for untreated wood. In fact, distinction is shown only in the rates of weight-loss results, which are from two to four times greater for untreated wood than for treated specimens.

Table VII indicates the relative effectiveness of treatment for all comparable lots of material of various thicknesses. With few exceptions, the time results are increased relatively more by treatment of thick specimens than by treatment of thin specimens, but the reduction in the rate of weight loss becomes less as thickness increases.

In the series of tests with built-up specimens referred to previously, it was found that the rate of loss decreases with increasing concentration for all thicknesses, but the time of flame penetration for specimens of the same thickness was increased only by heavy treatment.

(*b*) *Thickness.*—All time results increase as thickness of specimen is increased, but at a proportionately greater rate. Figure 6 shows the average relations for the time to reach 100 C. for two procedures and for the time of flame penetration for tests by procedure *E*. The total weight loss in grams also increases as thickness increases, but the relationship is different for untreated and treated wood. For thicknesses greater than about $1\frac{1}{4}$ in. the total weight loss is usually greater for treated specimens than for untreated specimens, probably because flame penetration occurs much later for the treated specimens of this thickness or over. The average relations between the nominal loss in weight and the rate of loss in weight, respectively, and thickness, are shown in Fig. 7. It is seen that the curves for treated and untreated wood diverge as the thickness decreases, particularly below 1 in., but somewhat differently for the two plots. The rates of loss for untreated specimens are distinctly separated from those for treated specimens, at least for thicknesses up to $1\frac{1}{2}$ in.

(*c*) *Density.*—An attempt was made to study the effect of density in the same manner as for the fire-tube results, but there were too few data for specimens of a given thickness to establish the relations quantitatively.

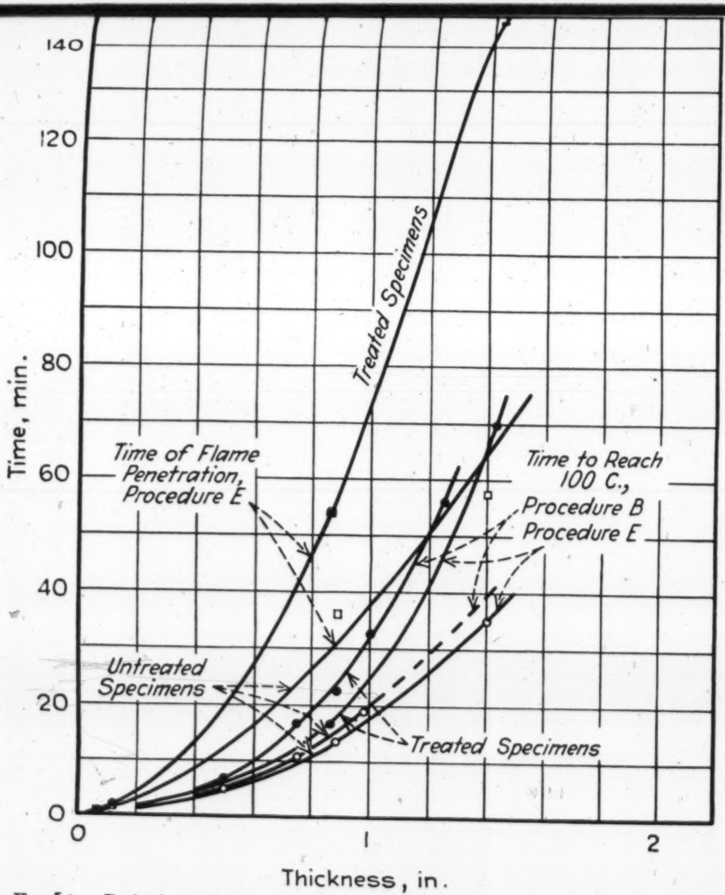

FIG. 6.—Relations Between Time to Reach 100 C., Time of Flame Penetration, and Thickness.

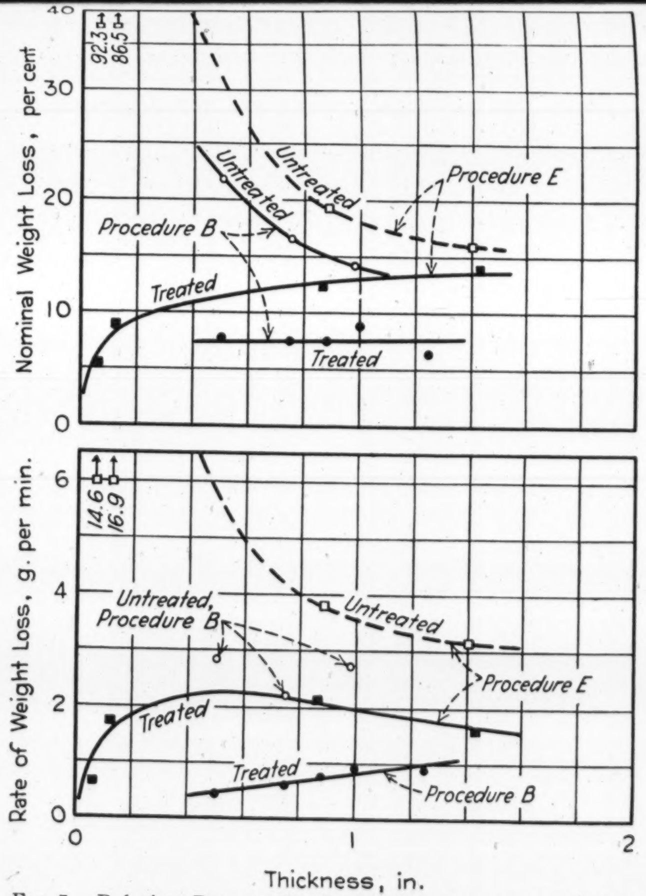

FIG. 7.—Relations Between Nominal Weight Loss, Rate of Weight Loss, and Thickness.

The time data seem to decrease slightly and the loss in weight in grams as well as the rate of weight loss quite definitely increase as density increases. The nominal loss in percentage does not appear to be affected by density.

(*d*) *Moisture.*—A general study was made of the effect of moisture using the individual data for ¾-in. specimens tested by procedure *D*, but the results are rather anomalous and the only conclusion that can be made is that, compared to species, moisture is only a moderate factor when in the range from 2 to 13 per cent.

TABLE VIII.—RESULTS OF SPECIAL STUDY OF EFFECT OF MOISTURE.

All ¾-in. Specimens Tested by Procedure *D*.

Specimen Data					Time to Reach 100 C., min.	Time to Reach 200 C., min.	Light Seen Through, min.	Flame Penetration or Ignition, min.	Net Duration of Flaming, min.	Loss in Weight		Rate of Loss, g. per min.
Species	Type[a]	Number	Moisture, per cent	Density, g. per cu. cm.						Actual, g.	Nominal, per cent	
SPECIMENS CONDITIONED AT 80 F. AND 30 PER CENT HUMIDITY												
Longleaf pine	U	2	6.4	0.66	10.3	14.8	16.4	17.2	15.9	223.2	27.2	6.76
Shortleaf pine	U	3	6.0	0.58	10.6	15.3	17.1	18.0	13.8	258.5	35.1	7.89
Sugar pine	T	2	5.5	0.50	14.0	24.4	24.4	28.0	0.2	64.0	10.4	1.94
Oak	T	3	4.4	0.69	12.1	24.9	27.9	36.0	0.5	81.7	9.5	1.96
Poplar	T	1	5.5	0.47	10.0	14.1	14.2	15.2	0.5	71.4	12.2	3.45
SPECIMENS CONDITIONED AT 70 F. AND 65 PER CENT HUMIDITY												
Longleaf pine	U	2	9.8	0.68	12.2	16.4	17.2	18.0	13.0	185.8	22.6	5.98
Shortleaf pine	U	3	11.0	0.59	12.4	19.0	22.7	26.3	13.2	231.9	30.0	5.49
Sugar pine	T	3	11.2	0.51	15.2	28.3	26.6	33.2	0.1	64.0	10.2	1.79
Oak	T	5	8.6	0.71	14.1	32.6	35.4	52.6	0.3	91.2	10.5	1.65
Poplar	T	2	11.9	0.50	13.0	18.4	18.4	20.3	0.2	68.8	11.4	2.69
SPECIMENS CONDITIONED AT 70 F. AND 100 PER CENT HUMIDITY												
Shortleaf pine	U	3	68.6	0.86	23.2	28.5	27.8	29.2	5.9	213.8	20.2	6.16
Sugar pine	T	2	97.3	0.93	38.0	45.6	45.4	53.0	0.0	261.4	23.8	4.60
Oak	T	3	68.0	1.07	34.6	50.6	49.3	61.3	0.0	333.0	25.3	5.08
Poplar	T	1	112.7	0.90	48.7	76.9	69.0	81.0	0.0	377.1	34.2	4.38

[a] U = untreated, T = treated.

In addition, a number of ¾-in. flame-penetration specimens were conditioned with the fire-tube specimens and tested at the same time. As can be seen in Table VIII, the average time of flame penetration increases with the moisture content. Although all of the weight loss data for the treated specimens are much greater when the moisture content is abnormally high (most of the weight loss is moisture) than when it is within the normal range, such is not the case for untreated specimens.

Summary:

These studies of the flame-penetration test indicate that:

1. Although the results for untreated specimens are more variable than those for treated specimens, it is believed that from three to five

specimens, properly selected, give average results representative of a given lot of material.

TABLE IX.—COMPARISON OF RESULTS BY THE FIRE-TUBE AND FLAME-PENETRATION TESTS ON THE SAME LOTS OF MATERIAL.

Material			Fire-Tube Results				Flame-Penetration Results				
Species	Source and Lot	Type[a]	Maximum Temperature, deg. Cent.	Loss in Weight		Maximum Rate of Loss, g. per min.	Thickness, in.	Time of Flame Penetration, min.	Loss in Weight		Rate of Loss, g. per min.
				per cent	grams				Actual, g.	Nominal, per cent	
							By Procedure *A*				
Oak	A-5	U	796	82	104.0	40.4	0.62	45.7	104.5	15.0	1.96
Western yellow pine	A-1	U	729	87	67.0	28.5	0.75	31.9	110.5	19.9	2.83
							By Procedure *B*				
Birch	C-5	T	163	14	20.6	6.2	0.50	26.4	42.9	6.9	0.48
White cedar	C-1	T	136	12	12.8	3.7	0.75	none	39.8	5.9	0.44
Chestnut	C-11	T	123	11	12.7	4.0	0.75	none	57.6	8.2	0.64
White cedar	C-2	T	165	14	14.9	4.4	0.75	none	48.6	7.2	0.54
Oak	C-4	T	136	13	13.6	4.9	0.75	none	55.2	6.9	0.62
Birch	C-7	T	154	15	21.4	6.3	0.75	34.4	65.9	7.5	0.73
Oak	C-3	U	662	82	83.0	32.5	0.75	61.5	126.6	18.4	1.86
Birch	C-6	U	693	86	118.1	39.4	0.75	39.9	139.0	15.9	2.89
Chestnut	D-5	T	163	18	15.4	4.6	1.00	68.0	68.7	8.8	0.76
Western yellow pine	D-1	T	167	21	17.1	6.0	1.00	75.4	85.1	11.2	0.94
Black walnut	D-6	T	141	14	18.2	6.0	1.00	63.4	118.4	9.9	1.32
Oak	D-3	T	172	18	26.9	8.1	1.00	80.1	113.4	10.1	1.26
Birch	D-4	T	228	25	32.8	11.5	1.00	49.1	96.3	8.5	1.07
							By Procedure *C*				
Black walnut	C-13	T	150	15	20.5	6.4	0.75	none	47.8	6.4	0.53
	C-12	U	792	84	90.8	31.7	0.75	49.8	90.2	14.3	1.64
							By Procedure *D*				
Longleaf pine	A-2	U	733	83	91.5	30.6	0.75	17.6	204.5	24.9	6.37
Shortleaf pine	A-4	U	756	84	95.3	31.6	0.75	26.3	231.9	30.0	5.49
	A-3	U	799	87	95.3	36.7	0.75	18.0	258.5	35.1	7.89
							By Procedure *E*				
White pine	E-2	T	172	19	15.8	5.3	0.81	74.5	98.2	13.2	1.28
Birch	E-4	T	204	22	31.2	10.5	0.88	30.4	110.9	10.4	3.06
Black cherry	E-13	T	398	48	55.4	15.2	0.88	34.7	103.8	10.8	2.60
Black walnut	E-9	T	418	46	60.6	17.3	0.88	76.5	120.6	14.6	1.64
White pine	E-1	U	732	81	56.8	24.7	0.88	36.0	118.8	18.4	3.21
Black cherry	E-12	U	722	76	80.6	31.1	0.88	33.4	124.5	15.4	3.32
Black walnut	E-8	U	733	82	97.6	34.7	0.88	51.5	185.8	25.2	3.09
Birch	E-3	U	728	82	99.3	35.5	0.88	29.9	190.9	21.2	5.24
Chestnut	E-6	T	182	18	22.5	6.0	1.06	69.9	131.8	12.3	1.80
	E-5	U	702	75	69.6	27.1	1.06	33.9	116.2	15.2	3.08
Black walnut	E-10	U	785	79	85.2	33.8	1.38	68.9	268.6	18.3	3.40
	E-11	T	638	47	53.3	17.8	1.44	194.0	327.3	17.1	1.73
Chestnut	E-7	T	322	33	33.8	11.3	1.78	178.0	238.4	12.6	1.30

[a] U = untreated, T = treated

2. The time of flame penetration appears to depend more on species than on the nature of the treatment. Some treated specimens may be no more resistant to flame penetration than untreated specimens from the same source. Consequently, treatment cannot be assumed as always

increasing resistance to flame penetration even though it does decrease combustibility.

3. In comparison with other factors such as species, the moisture content in the range from about 4 to 12 per cent seems to be a minor factor in the flame-penetration test.

4. The rate of loss is the most useful of the weight loss data, in that it distinguishes between treated and untreated wood of all thicknesses, and the difference between the values for these two classes of materials is quite constant for thicknesses above ¾ in. The rate of weight loss can be regarded as giving information on the intensity of combustion.

COMPARISON OF RESULTS

The more important results obtained by the two testing procedures on the same lots of material are compared in Table IX. A study of these data did not indicate any very definite relations between the several results obtained in one test and those obtained in the other. A relation between the maximum rate of loss in the fire-tube test and the average rate of loss found in the flame-penetration test might be expected, but it is not well defined, and appears to be different for different species.

The relative effectiveness of treatment in reducing the rate of weight loss affords another basis for comparison of the two tests. For nearly all lots of material tested by both methods and for which the flame-penetration specimens were ¾ in. or over, it can be seen that a relatively low ratio in the last column of Table VII corresponds to a low ratio in the last column of Table III, while a high ratio in one table corresponds to a high ratio in the other. Two exceptions are lots E-9 and E-11 of treated walnut, for which the individual results varied widely in both tests.

CONCLUSIONS

From the separate studies of the fire-tube and flame-penetration tests it may be concluded that either test can serve as a measure of the tendency of wood to support combustion and spread fire, and as an indication of the effect of treatment on these properties. The comparison of results, however, shows that data obtained in one test gives only an approximation of what would be obtained on the same material in the other test. The fire-tube test has the advantage that it permits a relatively sharper distinction between untreated, lightly-treated, and well-treated material, assuming proper selection of specimens. It is to be preferred when the samples are long enough to permit 40-in. specimens and where the equipment is available. The advantages possessed by the flame-penetration test are that no special equipment is required, shorter samples can be used in preparing specimens, and fewer specimens are needed since each specimen is representative of a larger cross-sectional area of wood.

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(29) T. R. Truax, "The 'Fireproofing' of Wood," *Proceedings*, Nat. Fire Protection Assn., Vol. 35, p. 189 (1931); *Safety Engineering*, Vol. 62, p. 179 (1931).

(30) J. F. Harkom and J. I. Dore, "Slow Combustion Test," *Proceedings*, Am. Wood-Preservers' Assn., Vol. 29, p. 87 (1933).

(31) A. T. North, "Trial of Fireproof Wood," *Architectural Forum*, Vol. 54, Part 2, p. 247 (1931).

(32) "Fire Tests of Partitions," *Red Book 193*, British Fire Prevention Committee (1914).

(33) "Fire Tests with Doors," *Red Book 194*, British Fire Prevention Committee (1914).

(34) Fire Test Shows Superiority of Wood Door Over So-called Approved Type, *Southern Lumberman*, Vol. 136, No. 1762, August 15, 1929, p. 63.

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(38) Standard Specifications for Fire Tests of Building Construction and Materials (C 19 – 33), 1933 Book of A.S.T.M. Standards, Part II, p. 254 (1933).

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(40) G. M. Hunt, T. R. Truax, and C. A. Harrison, "Additional Experiments in Fireproofing Wood," *Proceedings*, Am. Wood-Preservers' Assn., Vol. 27, p. 104 (1931).

(41) T. R. Truax, C. A. Harrison, and R. H. Baechler, "Experiments in Fireproofing Wood; Fourth Progress Report," *Proceedings*, Am. Wood-Preservers' Assn., Vol. 29, p. 107 (1933); also briefly in *Engineering News-Record*, Vol. 110, p. 591 (1933).

(42) T. R. Truax, C. A. Harrison, and R. H. Baechler, "Experiments in Fireproofing Wood; Fifth Progress Report," *Proceedings*, Am. Wood-Preservers' Assn., Vol. 31, p. 231 (1935).

(43) H. A. Fiske, *et al.*, "Report of Committee on Fire Retardant Treatments of Wood," *Proceedings*, Nat. Fire Protection Assn., Vol. 5, p. 64 (1901); also briefly in *Engineering News*, Vol. 45, p. 459 (1901).

(44) R. S. Kellogg, "Lumber and Its Uses," p. 150, Scientific Book Corp., New York City (1931).

(45) Dana Pierce, *et al.*, "Report of Committee on Status and Prospects of Fireproofing Processes," *Proceedings*, Nat. Fire Protection Assn., Vol. 36, p. 37 (1932); *Safety Engineering*, Vol. 64, p. 73 (1932).

DISCUSSION

Mr. S. R. Church.[1]—The author refers to tests on "well-treated wood." Since no particular description of the treatment or material used is given, I should like to know whether he could throw a little light on what he means by "well-treated."

Mr. C. R. Brown.[2]—In using the term "well-treated wood," I have been guided by the fact that the results of the Forest Products Laboratory show that for all of the treatments and concentrations they have studied, there seems to be a limit to the best that can be accomplished by treatment, corresponding to about 15 or 20 per cent weight loss in the fire tube. I have found that for most specimens treated commercially the loss in weight is 25 per cent or less, but when the weight loss of any specimen exceeded this figure, the maximum temperature often exceeded 250 C., indicating flame spread up the specimen towards the top of the tube. Consequently, I have arbitrarily used the term "well-treated" to designate material having a weight loss of 25 per cent or less, but I do not intend to infer that this should be used as a definite point for specification purposes.

Mr. A. S. Williams[3] (*by letter*).—This paper contains some interesting data and contributes to the general subject of testing of fire-retardant wood. With the increasing interest being shown in the subject of fire-retardant wood, it is fitting that work along this line be undertaken at the National Bureau of Standards. It is to be hoped that the Bureau will find it possible to continue the study of methods of testing fire-retardant wood on a more comprehensive scale.

The present paper seems to be open to two main criticisms. In the first place the work did not embrace a sufficiently large number of tests to justify forming dependable conclusions. In some cases results are given for only a single test and in others only two or three tests were made. The very nature of wood is such that, even when untreated samples are carefully selected and prepared for testing, considerable variation in test results are likely to occur. For this reason the number of tests should be sufficiently large to make sure that such irregularities have been properly provided for. This is particularly true when we are attempting to develop a test method, which I believe is the fundamental idea involved in this work. Where a standard method exists it is likely that such a small number of tests as used here may serve satisfactorily but in work such as described it would appear that at least five and probably ten tests should be made on each sample. Apparently most of the test material was used up in the development of the procedure and little left for testing under the finally adopted procedure. Table VIII, for example, gives the results of only 35 tests made upon five different species of wood under three different sets of conditions. It would seem that such a valuable part of the work would justify the performance of possibly five times this number of tests.

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[1] **Consulting Chemist, New York City.**
[2] **Assistant Physicist, Fire Resistance Section, National Bureau of Standards, Washington, D. C.**
[3] **Director of Testing Laboratory, Protexol Corp., Kenilworth, N. J.**

In the second place there is conspicuous absence of sufficient data covering tests on untreated and treated wood of the same species to enable one to make the comparisons which are so necessary in this kind of work. It seems that some species should have been selected as a control and a sufficient amount of material obtained so that at least this one species in both treated and untreated specimens could be followed through all of the tests. At least this should have been done in the case of tests made under the finally selected procedure, which in the case of the flame penetration test was procedure E.

The author has introduced a considerable number of variables which, in work of this kind, make it more difficult to interpret the results. Apparently some of the treated wood did not possess a high degree of fire retardance. This is particularly noticeable in some of the walnut, cherry and chestnut specimens. In order to compare test methods the test material should be uniform in properties. If variables are to be introduced the extent and kind of such variation should be known; otherwise serious errors are likely to occur. In most cases both the degree and nature of the treatment were unknown and this, together with the natural variation in the wood, introduces a strong possibility of inconsistency in results.

The author does not give data obtained through the use of thermocouples of various size wire in the fire tube test. Such data are important and if differences in temperature readings occur of a magnitude greater than those obtained ordinarily in a series of specimens from the same sample, a change in size of thermocouple wire would seem justified. It would be interesting to have these data.

The author speaks of poorly treated material without defining the term. What is probably meant is material of low fire-retardant value. A sample may be thoroughly treated with an inferior fire-retardant chemical or inadequately treated with an efficient chemical.

In giving the results of the fire-tube test, following Table I, the author states that hardwoods as a class do not necessarily give lower values for any of the results than softwoods. Such a conclusion would apply here only to untreated wood since too many variables or unknowns are involved in the treated wood to justify such a conclusion upon the data submitted.

The author makes use of the loss in weight in grams as a criterion. It would seem that the percentage loss is more valuable, together with the rate of loss.

In Table III the effectiveness or efficiency of the treatment, assuming uniformity of material and treatment, can be determined readily by taking the difference between the percentage loss in weight of the untreated and the treated material and dividing by the loss for the untreated. In this manner the treatments for specimens in lot B for longleaf pine are found to be approximately 80 per cent efficient.

In comparing the fire resistance of hardwoods and softwoods it is not correct to compare the percentage loss in weight for untreated materials in cases where exposure to the igniting flame is of sufficient duration to cause complete burning. In such cases only ash remains and the weight of the residue will be approximately the same for all ordinary species of wood. It follows then that for specimens of constant size or volume the weight loss will be greater for the more dense specimens. Therefore the total weight loss and probably the rate of weight loss will vary with density. A better means of measurement in such cases is the time to ignition or sustained burning. Even this might be affected by other factors such as resin content of the wood.

The information on the effect of moisture is interesting and would be even more valuable if more specimens were used and the species of wood had been kept constant. The fire resistance naturally increases with the moisture content, at least the time to reach sustained burning will increase. When the time of exposure is limited, then with increasing moisture content, the burning process gradually changes into a drying process. The moisture content must first be reduced to a certain point before sustained burning takes place. This is shown by the results given in Table IV. It is interesting to note that the weight loss in grams for treated longleaf pine remains practically constant over a moisture content range of 6.5 to 139.3 per cent. For the lower values, loss in weight is contributed to a greater extent by the actual destruction of the wood while at higher moisture content the loss is largely due to moisture or drying. Here again, comparison of untreated and treated material is prevented by not having the same species of wood.

In the flame penetration test the most important information obtainable is probably the time of flame penetration or rate of flame penetration. Such information for various thicknesses is very important from a commercial use standpoint. Where fire-retardant wood is used for fire doors and as a shield for covering metal girders, etc., this information becomes very valuable. Perhaps of equal importance is the time to reach various temperature stages such as 100 C., 200 C., etc. This might be used also as a measure of flame penetration. The other factor such as weight loss and perhaps rate of weight loss are dependent upon the time of burning or exposure and are therefore directly related to time of flame penetration.

The statement that the species is probably of more importance than the nature of treatment for treated specimens is certainly contrary to experience in the use of fire-retardant wood. Results obtained by the writer in a large number of tests of panels and of full-size doors do not substantiate the author's view. Many producers and users of fire-retardant wood will probably also disagree with this statement. British officials, for example, recognize a difference in the resistance of hardwoods and softwoods and require that softwoods be treated for certain locations where untreated hardwoods are permitted. It is likely that variations in treatment have caused the author to reach this erroneous conclusion. This indicates that in such tests material of uniformity in treatment and species should be used.

The data on the effect of thickness is also very interesting. It is noted that the resistance to flame penetration increases much more rapidly with increasing thickness in treated wood than in untreated wood. This clearly shows the effect of the treatment. The same holds true for time to reach 100 C. The results also show that at the temperature of exposure used in the tests a minimum thickness of approximately $\frac{1}{2}$ in. is necessary before the curve for rate of weight loss takes the expected form. This minimum thickness would probably vary with the intensity of the igniting flame.

A comparison of the methods shows an advantage in favor of the fire-tube test in all criteria that both tests are capable of measuring. The fire-tube test is far more sensitive and perhaps more dependable except for measuring flame penetration and temperature transmission which are not possible with it. The flame penetration test is probably limited to a measurement of these characteristics only.

It is recognized by most workers in this field that no single test is capable of supplying all of the information required for the proper evaluation of fire-retardant treatments for wood. The flame penetration idea is fundamentally sound and should be developed for measuring flame penetration and temperature transmission, both of which are very important.

MR. E. F. HARTMAN[1] (*by letter*).—One with a commercial interest in fire-retardant wood is very naturally interested in a yardstick that will best measure the fire-resistive qualities. Concordance in methods of evaluating results is vital to commercial progress. A method that is satisfactory to the experimenter may be of little value for comparing commercially treated woods. During 34 yr. the New York Building Department tests have been recognized and accepted as "standard" for measuring commercially treated fire-retardant wood. Proponents of new methods of testing must prove the present tests inadequate or unreliable before discarding the accepted methods.

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[1] President, Protexol Corp., Kenilworth, N. J.

Geo. L. Smith

Based on an experience of 28 yr. in directing a commercial research laboratory there is grave doubt in the writer's mind about accepting conclusions based on a very limited number of tests and with so many variables present. By using wood commercially treated by one process when comparing methods of testing we can eliminate one variable that may easily cause conclusions to be faulty.

The writer must confess to a feeling of regret that Mr. Brown has left out of consideration the progress in the development of the fire tube made in Germany. Schlegel[1] showed the need of shortening the tube in order to secure more accurate results. Morath[2] also found it advisable to use a shorter tube. Falck[3] referred to the fire tube and Professor Kristen of the Berlin Materials Testing Station discarded it altogether according to a communication from F. Moll who wrote the introductory to Schlegel's contribution. Doctor Moll also expressed his own opinion on the fire tube as follows: "This method is certainly very good for laboratory tests but it will never do for practical evaluations." In a paper presented before the Society a year ago[4] commenting on Mr. Brown's remarks, a table was presented on page 794 showing quite conclusively that the fire tube was not a satisfactory test for "commercially" treated wood. In addition to the data then presented our experience with the present fire tube test proves very clearly its failure to catch streaky treatments. Such streaky treatments are most often noted in poplar and what is known in the lumber trade as "sound wormy" chestnut. If we take a specimen of such chestnut from a board for the fire-tube test the chances are that the streaks will not be exposed in the fire, whereas in the crib test such streaky treatments are readily detected.

The author's choice of the terms "poorly treated material" and "proper treatment" is very unfortumate for one is at a loss to interpret them. In commercial practice one does not expect 100 per cent uniformity in the treatment of an organic material like wood. There are spotty or streaky and partial treatments. A 100 per cent saturation with a solution possessing little fire-retardant value could not be classed as a proper treatment. A partial impregnation with a solution of real fire-retardant value may be a proper treatment depending upon the desired fire resistance as in the case of scaffold timbers or planks.

May one inquire for the basis of the conclusion that little is gained by increasing the treatment beyond a certain concentration? The references cited all refer to experimental work by the U. S. Forest Products Laboratory on experimental and not commercial treatments. The nature of the impregnating solution or chemicals would appear to merit consideration.

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[1] R. Schlegel, "Untersuchungen über die Grundlagen des Feuer schutzes von Holz," Verlag Chemie Berlin (1934).

[2] E. Morath, "Anleitung zur Prüfung der Wirksamkeit chemischer Schutzmittel zur Herabsetzung der Brennbarkeit der Hölzer," Holzforschungsinstitut an der Technischen Hochschule, Darmstadt (1935).

[3] R. Falck, "Hausschwammforschungen," Heft 11 (1934).

[4] E. F. Hartman, A. S. Williams and R. C. Bastress, "Measuring the Combustibility of Fire-Retardant Wood," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 754 (1934).

In the tests on veneer specimens, these were nailed together. In commercial work veneers are glued. If we wish to know the fire resistance of built-up or veneered wood the glue must be considered.

In the summary on the fire-tube tests it is stated that: "At least five, and possibly ten specimens are necessary to obtain representative average results for a given lot of material . . ." In the summary on flame penetration tests, from three to five specimens are suggested. In commercial work we deal with lots running on an average from 100 board feet to let us say 60,000 board feet and they have run up to 500,000 board feet. How is one to determine on the number of specimens to be subjected to test?

The recent interesting fire on the forty-eighth floor of the General Electric Building in New York City might be cited as an instance for establishing the number of specimens to be tested. The stairbuilder will require one 12-in. wide plank 14 ft. long of 3-in. mahogany and possibly one hundred feet of 5/4 birch.

It is a pleasure to note the author's concurrence in our previous findings that the weight loss is a logical measure for evaluating results. To express this as a percentage loss together with the rate of loss would appear most rational. Unfortunately this is not correct for the fire-tube test where only 25 per cent of the specimen is exposed to the flame.

Conclusion 2 of the summary on flame penetration tests is diametrically at variance with findings of the writer's laboratory. The conclusion is also at variance with the long-established rules of the London Building Acts which have the sanction of the British Fire Prevention Committee.

Flame penetration tests appear likely to deserve further attention since these come nearer to performance tests, and resistance values are so desirable for evaluating building materials. In the writer's laboratory a small furnace is used in which one can test on the time-temperature curve. Panel tests are exclusively required by the German Government.[1]

It may be of interest to note that in Germany the National Government issues licenses to those engaged in giving fire-retardant wood treatments based on tests conducted at the Government Testing Stations at Berlin, Dresden or Stuttgart. Only licensed processes may be offered for use.

MR. BROWN (*author's closure by letter*).—One of the principal reasons for making these studies has been to supply information on the fire-tube and flame-penetration tests to those engaged in preparing specifications for testing methods, such as the Society's Committee C-5 on Fire Tests of Materials and Construction, in the belief that no method had as yet been accepted as adequate or standard throughout the United States. This belief was strengthened by a study of the literature, although it was apparent that some of the earlier methods might be developed into useful procedures. We have confined our efforts to two methods, leaving others to other investigators, and we wish to present here the advantages and limitations of these methods as we see them, leaving the final choice of standard methods to those charged with their selection.

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[1] Belastungen und Beanspruchungen im Hochbau (1935); also Prof. Dr-Ing. Kristen und Prof. Dipl-Ing. Schulze, Bericht über Versuche mit geschütztem und ungeschütztem Holz bei Feuereunwirkung, *Der Bauingenieur*, Heft 21/22 (1935).

The scope of an investigation is obviously limited so that an arbitrary choice of procedure must be made in order to make the best use of the time and material available. We have felt that some 400 fire-tube and 200 flame-penetration tests were sufficient to warrant the conclusions we have made. Variables other than the factor being studied have been eliminated or reduced in importance wherever possible, and where the number of tests does not permit definite conclusions, we have so stated in the text. Moreover, studies of the results for individual species and treatments did not develop any conclusions contrary to those made for treated and untreated wood in general. We do not agree that the tests should have been confined to one species and one treatment, since conclusions based on the results would not necessarily apply to other species and treatments. We thought it best to rely mostly on average results of a limited number of tests on each lot, so as to include several species and treatments of both the experimental and commercial type. Since information on the nature of proprietary treatments has been excluded from the tables, we have used only experimentally treated specimens of known concentration and treatment in our studies of these important factors. Moreover, our results do not show that the best commercially treated specimens were sufficiently different (if at all) from the best experimentally treated specimens to warrant the exclusive use of either in making these studies.

We would like to have included a more detailed discussion of the literature on testing methods, but space did not permit this. Regarding Mr. Hartman's references to recent German studies, unfortunately most of the literature cited was not available to readers in Washington at the time of preparing the paper.

By the term "poorly-treated material" we have meant either wood treated with light concentrations, or wood in which the distribution of chemicals was poor. In either case the effect on fire-tube results was the same, that is, relatively high losses in weight and high maximum temperatures. Treatments known to have little or no fire-retardant effect were not included in these tests. The term "proper treatment" means any suitable treatment which produces the required fire-retardant effect. As indicated in the author's reply to Mr. S. R. Church's question, there is apparently a limit to the extent to which the weight loss, etc., can be reduced.

Our tests on specimens built up from veneer stock were made merely for the purpose of determining the suitability of the test for lumber less than the thickness of $\frac{3}{8}$ in. required for solid specimens. We were not testing the construction itself, and nailing seemed to be satisfactory for our purpose.

The maximum temperatures for treated specimens averaged 11 deg. Cent. higher when fine-wire couples were used than when the No. 14 gage couple was used, although the weight loss and rate of loss averaged the same in both series of tests, the materials being the same in both cases. Greater differences would be obtained for untreated wood.

In suggesting the number of specimens to be prepared from a sample, we assume that the sample itself shall consist of a number of boards carefully selected from a lot of material, the size of the lot and the manner of selection to be defined in well-considered specifications of the material.

As discussed in the paper, each of the fire-tube results seems to have a particular meaning, so that the value of each as a criterion depends in part upon the information desired. In questioning our use of the weight loss in percentage as a basis for comparing different lots of untreated wood, Mr. Williams infers that the fire tube permits complete combustion of untreated wood to ash. This is hardly true since the ash content of most woods is less than 1 per cent and the weight of the untreated specimen after a fire-tube test is from 13 to 25 per cent of the original weight. Furthermore, our comparisons of fire-tube results are based on other results as well as the weight loss. As Mr. Williams points out, the statement that "hardwoods as a class do not necessarily give lower values for any of the results than softwoods" applies only to untreated specimens, since other factors are involved in tests of treated specimens.

A simple calculation converts the ratios given in Table III to the ratios suggested by Mr. Williams as a measure of "efficiency of treatment." Either figure is suitable.

Contrary to Mr. Williams' expectations we did not find that the weight loss and rate of loss are always related to the time of flame penetration. For untreated wood, no relations could be observed at all, while for treated wood, the weight loss in grams or percentage seems to increase very slightly with increasing the time of flame penetration. For treated wood, the rate of weight loss actually decreases as the time of flame penetration increases.

Both Mr. Hartman and Mr. Williams take exception to our second conclusion from the flame-penetration tests. There appears to be sufficient evidence in the tables that the time of flame penetration of either treated or untreated specimens varies more widely when species is the only factor than it does between treated and untreated specimens of the same species. Also, while treated specimens are usually appreciably more resistant to flame penetration than untreated specimens there are some exceptions to this rule, which cannot be charged to variations in treatment since the rate of weight loss figures indicated well-treated material. The statement that British officials make a distinction between hardwoods and softwoods does not appear to be inconsistent with our results in the flame-penetration tests. With respect to fire-endurance tests, it may be of interest to note that we found very little difference between the fire endurance of treated and untreated longleaf pine panels of the same construction and thickness.[1]

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[1] C. R. Brown, "Fire Tests of Treated and Untreated Wood Partitions," publication pending.

TESTING THE PHYSICAL AND CHEMICAL PROPERTIES OF WOOL BY MEANS OF THE MICROSCOPE

BY WERNER VON BERGEN[1]

SYNOPSIS

The main purpose of this paper is to establish a basis to further the wool standardization work of the Society by reviewing the modern microphysical and microchemical tests used in studying and testing wool fiber.

To this end there is given:

1. A brief description of the structure of the wool fiber and the different layers as seen with the help of photomicrographs.
2. Two methods of measuring the fineness of wool and a key for quality determination.
3. A method for qualitative and to a certain extent quantitative determination of sunlight damage as well as hydrogen peroxide and chlorine damage.
4. Two methods for determination of alkali damaged wool.
5. A description of acid damage.
6. A brief description of mold in wool and the injury done by bacteria growths on wool fibers.

INTRODUCTION

The foundation of the wool manufacturing industry is the correct judgment of the quality of the wool. For hundreds of years this judgment was based mainly on visual inspection. The insufficiency of earlier methods was realized when the synthetic fibers started to conquer the world. This success was largely due to the thoroughly scientific understanding of their properties found through systematic research.

During the last decade an extensive research in the wool industry has set in all over the world. The fruit of this research has been condensed and may be found in the books of Mark,[2] Barker,[3] Froelich, Spoettel and Taenzer[4] and Kronacher.[5] Today, we have a much more scientific understanding of the properties of wool and their influence in the manufacturing of quality woolen goods.

Since the wool fiber is of microscopic structure, the use of the microscope is absolutely essential in finding some of the physical and chemical characteristics, which have been discovered by this research.

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[1] Chief Chemist, Forstmann Woolen Co., Passaic, N. J.

[2] H. Mark, "Beiträge zur Kenntnis der Woole und ihrer Bearbeitung," Gebrüder Borntraeger, Berlin (1925).

[3] S. G. Barker, "Wool Quality," His Majesty's Stationery Office, London (1931).

[4] G. Froelich, W. Spoettel and E. Taenzer, "Technologie der Textilfasern," *Woolkunde*, Vol. VIII, Part 1 (1929), issued by R. O. Herzog, published by Julius Springer, Berlin.

[5] C. Kronacher and A. Lodemann, "Technik der Haar- und Wooleunter-suchung," Urban und Schwrazenberg, Berlin (1930).

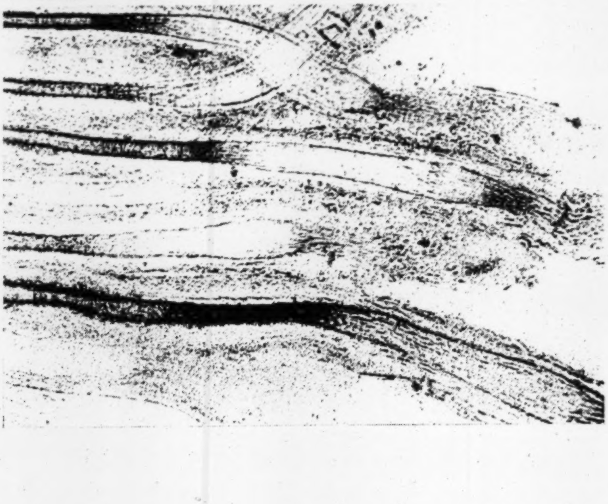


FIG. 1.—Wool Roots in Skin Pieces (× 80).

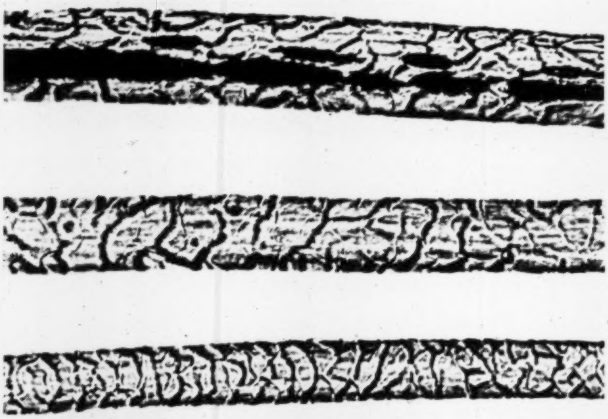


FIG. 2.—Coarse Wool Hairs, a Medullated Fiber (× 240).

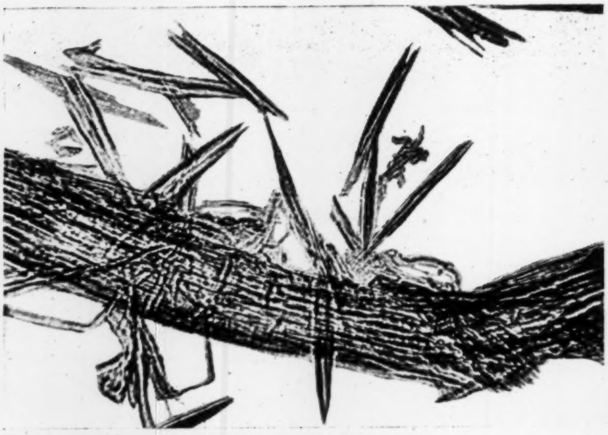
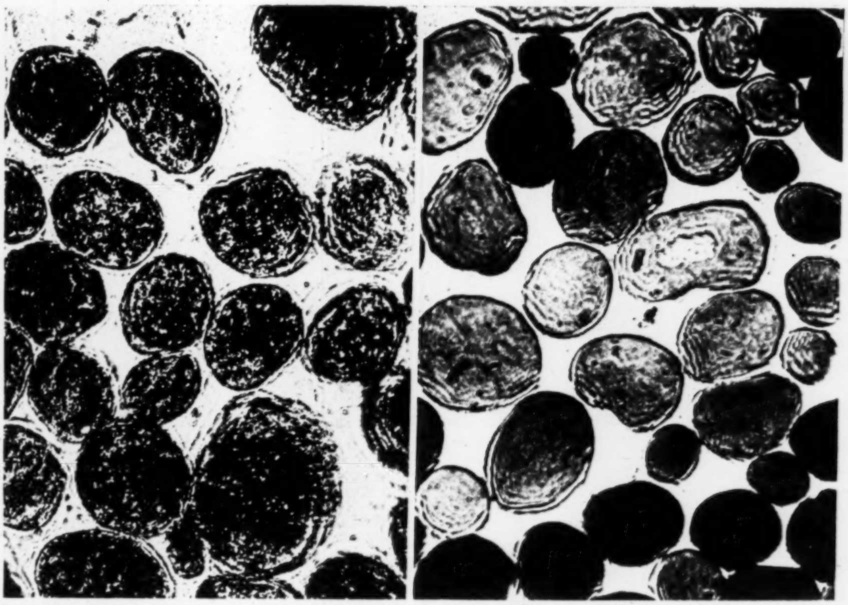


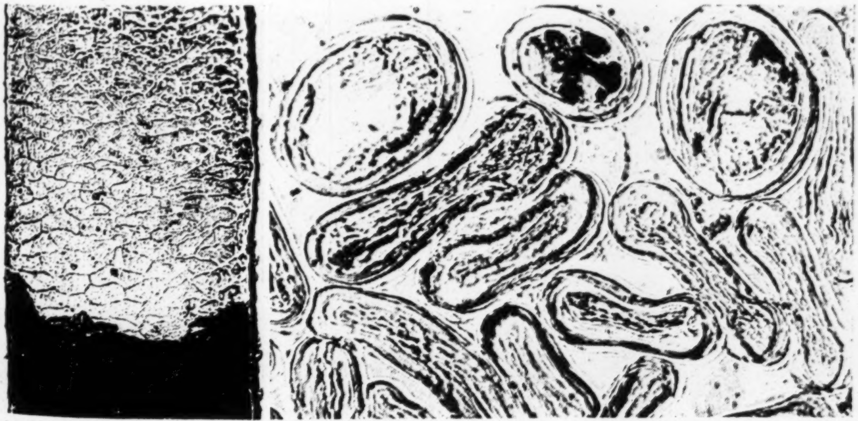
FIG. 3.—Wool Hair with Free Cortical Cells, Epidermis Destroyed (× 240).



(*a*) Dark brown domestic wool showing the distribution of the colored pigments in the cortical layer (× 500).

(*b*) Artificially dyed black wool, showing no pigmentation (× 500).

FIG. 4.—Cross-Section of Wool



(*a*) Kemp hair nearly 100 per cent medulla. In the black part of the fiber the medulla cells are filled with air (× 120).

(*b*) Cross section of kemp hairs showing their various forms, circular and ribbonlike. The black parts in the medulla are air-filled cells (× 250).

FIG. 5.—Kemp Hairs.

The members of the Subcommittee on Wool of the Society's Committee D-13 on Textile Materials, having set up tentative standards for woolen and worsted yarns, came to the conclusion that the highest value of these could not be realized until standards for the wool fiber itself had been set. This paper is presented as a basis upon which essential standards for wool fibers can be developed by the committees of the Society.

STRUCTURE OF THE WOOL HAIR

Wool is a variety of hair forming the protective covering of sheep. As a product of the skin it is of similar origin and fundamentally the same as horns, nails and hoofs.

Wool is an organized structure growing from a root situated in the dermis or middle layer of the skin (see Fig. 1), its ultimate physical elements being several series of animal cells of different forms and properties.

In the highest breed, the merino wool, the fiber consists of two portions:

(*a*) The outer layer, or epidermis, made up of flat horny cells or scales, the ends of which generally overlap each other and project outward, causing the edge of the fiber to present a serrated appearance under the microscope (see Fig. 2).

(*b*) The corticle, forming the main part of the stem, consisting of long, stretched, spindle-like cells, bound compactly. On account of this length formation the hair presents longitudinal striations which are visible through the epidermis. The average sizes of the cortical cells are 0.08 to 0.11 mm. long, 0.002 to 0.005 mm. broad, and 0.0012 to 0.0016 mm. thick (see Fig. 3). The cortical layer gives the fiber its chief strength and elasticity.

In coarse wool, a third layer is often present, a cellular marrow or medulla found in the axis of the hair. It is built up of many superimposed cells, of various forms, often polygonal, like a honeycomb. The diameter of the cells varies from 0.007 mm. to 0.001 mm. Various porous channels pass through the medulla cells, which are normally filled with air, as seen in Figs. 2 and 5 (*a*), longitudinal view, and in Figs. 4 (*b*) and 5 (*b*), cross-section.

In natural colored wool the color is caused by dyestuff pigments present in the cells of the cortical layer as well as in the medulla. This can clearly be seen in Fig. 4 (*a*). For comparison in Fig. 4 (*b*) we have a cross-section of black wool dyed with a combination of coal-tar dyestuffs with the assistance of sulfuric acid.

The shape of the cross-section of the wool fiber varies greatly, as can clearly be seen in Figs. 9, 10 and 11. Although some are nearly circular in shape, as a rule they are irregular and have a varying degree of ovality. The medulated fibers are clearly recognized by the black ring in the middle of the fiber.

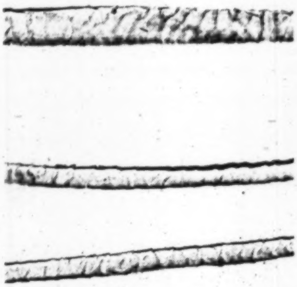


FIG. 6.—80's Wool (× 160).

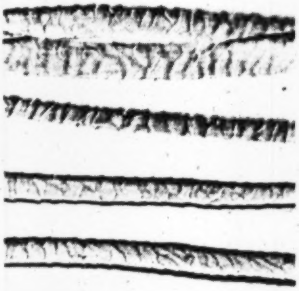


FIG. 7.—64's Wool (× 160).

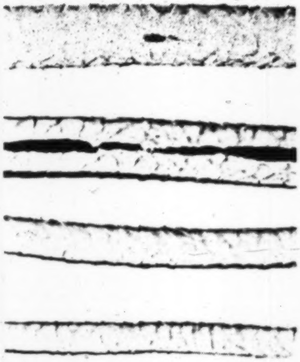


FIG. 8.—36's Wool (× 160).

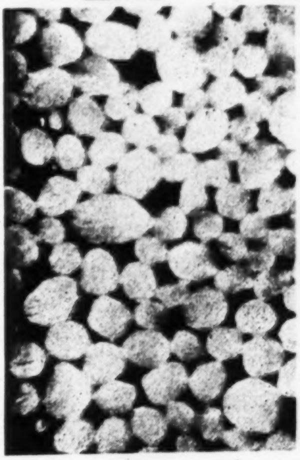


FIG. 9.—Cross-Section of 64's Wool (× 180).

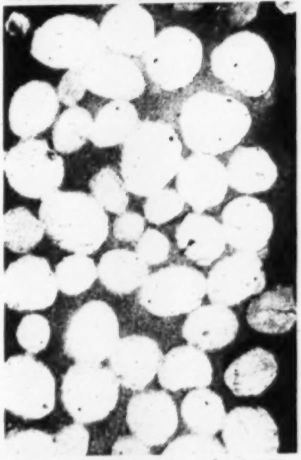


FIG. 10.—Cross-Section of 56's Wool (× 180).

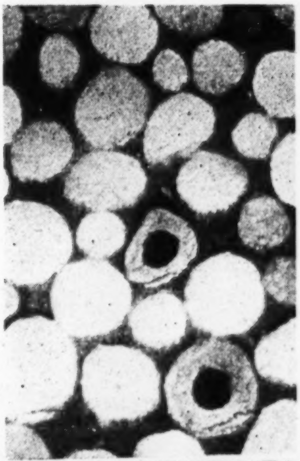


FIG. 11.—Cross-Section of 36's Wool (× 180).

In kemps, the medulla forms 90 per cent or more of the fiber. As with other fibers, only the cortical layer absorbs the dyestuff; therefore the kemps are much lighter in color when dyed together with normal fibers. Kemp fibers often have a ribbon-like form as seen from the cross-section of a kempy wool, Fig. 5 (*a*) and (*b*).

FINENESS OF WOOL FIBERS

The average fineness of the wool fibers is a dominant dimensional characteristic of the material immediately affecting its value for manufacturing purposes. One of the principle problems of the wool research was to find a quick and accurate method of judging the fineness.

Sheep do not produce wool in a condition immediately suitable for yarn requirements, each fleece carries strikingly different qualities of wool, and no two fleeces, even from the same type of sheep from the same producing district, are exactly alike in quality and quality distribution. If the manufacturer were to use the fleece in its original form he could only produce coarse and uneven yarns.

From these facts it is obvious that prior to processing, the fleeces must go through a sorting process. Sorting, therefore, consists of subdividing a fleece into its different qualities, the number of "sorts" made depending on the character of the fleece and the standard of yarn required. In present commercial practice the routine grading of wool is done by men of long experience in the industry who by merely handling and observing the material assign it intuitively to its proper grade. Such an estimate has many sources of error principally based upon mental and physical qualities of the sorter. At the same time other factors, as for example light or color and luster of the hairs, considerably influence the result. Through experience we know that by a weak light the estimate is too fine and by direct sun it is too coarse. Because of all these disadvantages, for more than one and a half centuries the replacing of the system of manual sorting by a system of measurements in a scientific manner has been felt necessary.

The main difficulty met in the way of establishing a scientific method is the fact that the diameter of the wool fiber varies between 10 and 30μ in the finest wool and from 10 to 70μ in coarse wool. Each fiber varies, also, in its thickness over the whole length, and, as the cross-section proved, the fibers are not circular in shape. The variation of the width in each grade is illustrated in Figs. 6, 7 and 8 and the variation in the diameter (cross-section) in Figs. 9, 10 and 11.

In July, 1926, the United States Department of Agriculture promulgated the Official Standards of the United States for Wool and Top.[1] After the adoption of this standard, the scientist had a foundation upon which to build an accurate method of measurement.

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[1] U. S. Department of Agriculture *Service and Regulatory Announcements No. 100*, July, 1926.

The most astonishing results were found by Winson[1] as he carried out a painstaking research on the measurement of the standard top. He found that for a range of selected British tops (the same tops were used in making up the U. S. master set) the progressive scale of fineness ascending from 48's to 80's quality is in geometric progression. A similar relationship was found for the French, German and Italian standard tops and it proved that the fundamental basis underlying wool sorting was the same in all countries.

In 1860, Fechner in his "Elements der Psycho-Physik" put forward the law now known as the Fechner-Weber Law, which states: "In order that the intensity of a sensation may increase in arithmetical progression the stimulus must increase in geometrical progression." This law, holding good between certain limits only, is expressed in his general formula $I = C \log S$, where I represents the sensation, S the stimulus and C is a constant. If we regard the wool sorter's judgment as indicative of I, then it must immediately follow that any attempt on his part to form a gradation of finenesses will result in a scale in which successive finenesses increase in geometrical progression. This is exactly the case in practice in all countries.

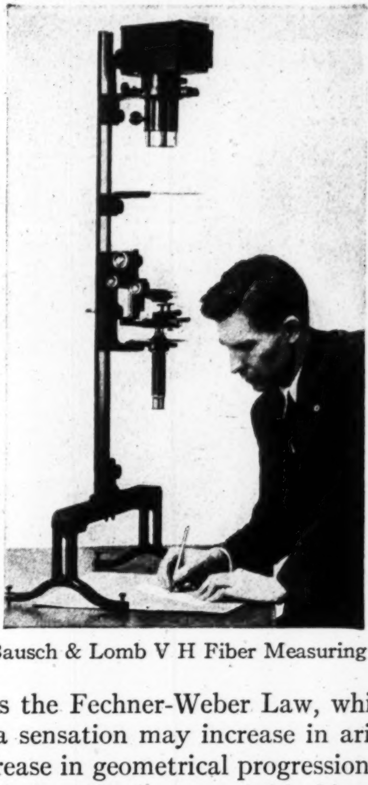

FIG. 12.—Bausch & Lomb V H Fiber Measuring Apparatus.

It can be said that for the normal operation of the wool sorter the

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[1] C. G. Winson, "Fiber Fineness and Wool Quality in Combed Tops," and "A Comparison of the Fineness of British and Continental Standards for Combed Tops," *Journal*, Textile Inst., Vol. 22, pp. 143 and 539, respectively (1931).

Fechner psycho-physical law is the fundamental basis of this work; therefore, for wool sorting the eye in the visual sense is the paramount factor.

MEASUREMENT METHODS

The main achievement resulting from the enormous amount of research on wool measurement is the fact that today we have three methods suitable for fiber examination:

1. Width method,
2. Cross-section method, and
3. Gravimetric method.

The equipment and technique required for the use of these methods will be described at this point.

Width Method:

The old way of measuring the width of fibers through a microscope with a micrometer is too tedious and not accurate enough, and it has been discarded. Today, the method has as its basis the projection of the image of the fiber, at a high magnification, through a microscope onto a screen. The width of the image of the fiber is then measured with various devices. The best device, by far, is to record the width of the image on a wedge ruler by which the hairs are automatically sorted according to their width. The method is as follows, using the apparatus especially built for this purpose, the Bausch & Lomb V H Fiber Measuring Apparatus, which was assembled on the basis of the author's[1] experience (see Fig. 12):

The slides are prepared by mounting about 100 fibers across the slides in glycerine and then covering them with cover glasses, the slide is placed on the upper side of the stage and the mechanical stage is adjusted to bring the slide in line with the objective, so that when using the sideway motion each fiber can be brought successively into view at approximately its middle point.

The starting point is at the right end of the cover glass moving to the left end in a straight line during the measuring.

The most important part of the method is the recording of each measurement on a wedge ruler (Fig. 13), a sheet of white cardboard 2 in. wide and 10.5 in. long. The inclination of the two lines towards each other is 1:20. On one end of the card the place where the two lines are exactly 5 mm. apart is marked with a crossline. As the wedge ruler is based on a magnification of 500, this space of 5 mm. must be divided by 500 to get the actual size, which is 0.010 mm. or 10μ.

At a distance of 200 mm. from the first crossline there is another crossline marking the place where the space between the two lines is 15 mm. which is equal to 30 μ. On the reverse side of the card the wedge ruler is printed for fibers with a width of 30 to 50 μ. By the use of this wedge ruler we are able to enlarge the magnification 20 times and we actually get a magnification of 500 × 20 = 10,000. The measuring of the fiber is performed in the following manner:

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[1] Werner von Bergen, "Measurement of Fiber Widths by the Wedge Method," *Melliand Textile Monthly* Vol. IV, Nos. 3, 4, 7, 8 (1932).

The wedge ruler is so placed that the image of the fiber is projected directly between the two lines of the wedge. The place where the wedge has exactly the same width as the wool fiber is marked with a short pencil line. The crossing points of the left and right edges of the image with the two lines are clearly recognized. The photomicrograph (Fig. 14) illustrates the recording in a clear way. A numerical reading is not necessary and therefore reading mistakes are eliminated.

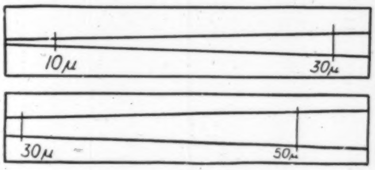

FIG. 13.—Wedge Ruler.

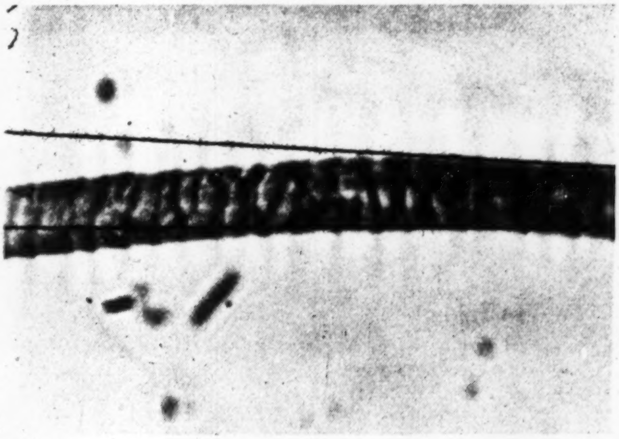

FIG. 14.—Photomicrograph Showing Recording of Width by the Wedge-Ruler Method (× 500).

For one analysis 100 hairs are measured and accordingly 100 pencil lines are drawn, which can be marked either in the middle or on the left or right of the wedge ruler. Every time a hair is measured the measuring is registered on a counter operated by a foot pedal to avoid unnecessary counting. By this method the hairs are automatically sorted according to their width. The final results show the true variation of the hairs within the samples.

With some practice one person is able to measure 100 fibers in 15 min.

From the wedge markings the fineness of the fibers can be calculated and specified in terms of the arithmetic mean width in microns and other methods representing dispersion. By simple comparison of the wedge ruler bearing the pencil lines marked up with a set of standards it is possible to judge the fineness of a sample very quickly and accurately enough for many mill purposes.

The most practical way to find the arithmetic mean and the distribution is by dividing the wedge, after the recording of the 100 fibers, into classes of 2.5 μ by putting over it a transparent ruler. By counting the pencil marks in each of the classes we get the percentage directly, because 100 fibers were measured.

The method was applied in measuring two sets of the Official Standard of the United States for wool top. The result derived from 500 measurements of each grade of the top are listed below:

QUALITY	MEAN, μ	STANDARD DEVIATION, μ
80's	19.5	4.20
70's	20.8	4.12
64's	21.9	4.67
60's	23.5	5.53
58's	24.8	5.45
56's	27.0	6.90
50's	30.4	7.20
48's	33.0	8.17
46's	34.8	8.05
44's	36.6	9.18
40's	38.3	8.30
36's	39.8	10.13

To study the accuracy of the method further, the average of the mean of five samples of 100 fibers and the standard deviation of the average of these five samples was calculated, and in addition the 68.3 per cent range, as the data were obtained under controlled condition.

QUALITY	AVERAGE OF MEAN OF 5 SAMPLES, μ	68.3 PER CENT RANGE, μ
80's	19.5 ± 0.52	19 to 20
70's	20.8 ± 0.68	20.2 to 21.5
64's	21.9 ± 0.66	21.2 to 22.5
60's	23.5 ± 0.76	22.7 to 24.2
58's	24.8 ± 0.99	23.8 to 25.8
56's	27.0 ± 0.36	26.5 to 27.3
50's	30.4 ± 1.28	29.0 to 31.6
48's	33.0 ± 0.50	32.5 to 33.5
46's	34.8 ± 1.19	33.6 to 36.0
44's	36.6 ± 1.20	35.4 to 37.8
40's	38.3 ± 0.60	37.7 to 38.9
36's	39.8 ± 1.49	38.1 to 42.1

The number of fibers which should be measured in order to be sure that the results indicate the true average of the sample depends on the type of material. For top, roving, and yarn, the author found that 200 fibers taken from two different parts of the sample gave satisfactory results.

When this method is used for checking up mill samples, even 100 fibers are sufficient.

When a practical spinner has to decide on the quality of a wool he usually looks for the number of coarse hairs present, and from the approximate percentage of the coarse hairs he bases his decision as to the quality of the wool. In order to put this custom on a more scientific basis, the author finally developed a key which enabled him to determine the quality of any wool sample accurately. The key is shown in Table I.

Cross-Section Method:

As mentioned previously, the shape of the wool fiber varies greatly and it is therefore a matter of great importance that a method for measuring contour and cross-sectional area should be available. The first thing to be considered is a suitable method for mounting the fibers for microscopical examination. The method followed by advanced workers in botany, biology, and similar fields, consisting of embedding the specimen in paraffin and then cutting them on a microtome, is the most precise, but it is too slow and too expensive to be applied to routine work on textiles. This has led to the development of several rapid methods giving satisfactory results.

TABLE I.—UNITED STATES STANDARD OF WOOL TOPS, KEY FOR QUALITY DETERMINATION.

QUALITY	AVERAGE, μ	PERCENTAGE OF HAIRS COARSER THAN 30 μ	40 μ	50 μ
80's	(19 to 20.5) 19.5	1 to 3		
70's	(20 to 21.5) 20.8	2 to 5		
64's	(21 to 23) 21.9	5 to 10		
60's	(22.5 to 24) 23.5	10 to 15	0 to 2	
58's	(23.5 to 26) 24.8	15 to 25	1 to 3	
56's	(25.5 to 28.5) 26.9	25 to 40	3 to 6	
50's	(28 to 31.5) 30.4	40 to 60	5 to 15	
48's	(31.5 to 34) 33.0	over 60	15 to 20	0 to 3
46's	(33.5 to 36) 34.8	......	20 to 30	2 to 5
44's	(35.5 to 38) 36.6	......	30 to 40	3 to 8
40's	(37 to 39) 38.3	......	40 to 50	5 to 10
36's	(38 to 42) 39.3	......	over 50	10 to 15

(*a*) *Metal Plate Sections Method.*—Holes $\frac{1}{32}$ or $\frac{1}{64}$ in. in diameter are drilled through a metal plate or dish. A small bundle of fibers are placed in the loop of a fine wire and drawn through the hole of the metal condenser or cross-section mount. The fibers must fill the hole completely to insure their remaining in place during the cutting. The actual cutting is done with a sharp razor blade, first the fibers on the bottom side of the plate are cut in one clean sliding stroke and then the fibers projecting from the top of the plate are cut in a similar manner. This technique, developed by Schwarz[1] has been found to be very useful, especially for rayon fibers. Hardy[1] has developed an improved cross-section device in connection with his work in studying the fineness of wool. This device, built of hard metal, is illustrated in Fig. 15.

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[1] E. R. Schwarz, "Textiles and the Microscope," McGraw-Hill Book Co., Inc., New York City (1934).

Both the fiber holder and slide holder are $\frac{3}{64}$ in. (1.19 mm.) in thickness. The shape of the fiber holder and slide holder when assembled is that of an ordinary microscope slide, 1 in. (25.4 mm.) wide and 3 in. (76.2 mm.) long. The fiber holder and slide holder are each $1\frac{1}{2}$ in. (38.1 mm.) long. The width of the fiber slot is 0.0085 in. and its length is $\frac{3}{8}$ in. The Hardy method of procedure is as follows:

A small lock of wool fibers is cleaned with carbon tetrachloride and dried between blotting paper. From this is separated a strand of fibers, about 1 mm. in diameter and containing usually over 100 fibers. This strand of wool is inserted in the fiber holder and the slide of the slide holder is pushed into the fiber slot until the strand of fibers is held securely in place. The fibers are then cut off flush on both sides of the fiber holder by means of a razor blade. With a little practice and sharp razor blades, good cross-sections are easily cut in less than one minute. Best results are secured by observing the position of the razor blade, while cutting, by means of a binocular microscope magnifying about 12 diameters.

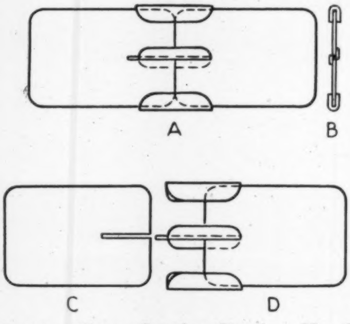

FIG. 15.—Cross-Section Device, Hardy.

(*A*) Top plan view of the whole device.
(*B*) End plan view of slide holder.
(*C*) Top plan view of the fiber holder disk showing the fiber slot.
(*D*) Top plan view of the slide holder.

Should the fibers appear to be packed too tightly, the slide holder is withdrawn until the individual cross-sections of the fibers stand out in relief when examined under the microscope. The cutting of very fine fibers may be facilitated by allowing a thin solution of celluloid in acetone to dry on the fibers in the fiber holder before cutting.

The cross-section device is placed on the stage of a projecting microscope (Bausch & Lomb's V H Fiber Measuring Apparatus is especially suitable) and the cross-section is focused upon a sheet of white paper in exactly the same manner as described under the width method.

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[1] J. I. Hardy, "Determination of Fiber Fineness and Cross-sectional Variability," *Textile Research*, Vol. 3, December, 1933, p. 381; Vol. 5, February, 1935, p. 184.

The projected images of the cross-sections may be measured as projected or photographed by projecting on a sensitized paper and measured after developing. If measured in the dark room the paper-wedge measure used by Lauth[1] and von Bergen is most satisfactory, inasmuch as the diameter distribution curve of the fibers is obtained as the diameter of each fiber is recorded.

Photomicrographs of cross-sections of fibers are measured with greater convenience and less fatigue than their images as projected in the dark room.

One hundred fibers are measured in two directions, the major and minor axes. From the results, the average mean and the standard deviation is calculated. By making standard photographs of the cross-sections of the standard wool fibers, a quick grading of any sample of wool can be made by direct visual comparison. The three photomicrographs (Figs. 9, 10 and 11) illustrate this point.

(*b*) *Cork Method Sections.*—This method was worked out first by Viviani and improved upon by Herzog.[2] A fine crochet needle is forced through the center of a small cork stopper of the best quality. On the projecting hook of the needle is laid a strong silk yarn and the needle together with the thread is carefully withdrawn until a fair-size loop of the silk is pulled through. Through this loop the fiber bundle is drawn and by withdrawing the silk thread the bundle of fibers is pulled through the cork in the same manner as they were drawn through the metal plate. The cork can be put in a microtome and cross-sections made in the usual way.

(*c*) *Rapid Paraffin Section Method.*—The laboratories of the Wool Industries Research Association, England, have adopted a simplified method of embedding the fibers in wax or paraffin, but this method, described by Barker,[3] is still too long. Herzog[2] has developed a quick technique by successive dipping of a bundle of fibers in melted paraffin, and building up a small candle of paraffin from $\frac{1}{16}$ to $\frac{1}{4}$ in. in diameter containing the fibers in the same manner as a wick in a candle. After hardening, the candle can be cut in the regular manner.

Of these three rapid methods of preparing cross-sections, the metal plate section is the most used for the examination of textile fibers. The rayon subcommittee of the American Association of Textile Chemists and Colorists has adopted it as the standard method for rayon fiber identification. The wool subcommittee of the Society's Committee D-13 onTextiles will probably adopt the technique of Hardy for diameter determination of wool fibers and issue it as a tentative standard.

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[1] E. Lauth, "Neue Mittel zur Feinheits-Längen- und Kraftbewertung von Kamm-Wollen," Dissertation, Munich (1930).

[2] P. Heerman and A. Herzog, "Mikroskopisch und Mechanisch Technische Textiluntersuchungen," p. 70, Julius Springer, Berlin (1931).

[3] S. G. Barker, "Wool Quality," p. 114, His Majesty's Stationery Office, London (1931).

Gravimetric Method:

This method consists of weighing a definite number of fibers cut to a certain length and expressing the mean fineness in terms of the weight of a standard length at a standard regain.

The International Wool Organization has adopted as its unit of fiber fineness the weight in milligrams of 10 meters length of wool fibers at a regain of 18¼ per cent. As this method, which was worked out by Winson, is non-microscopical it will not be discussed further.

Which of these three methods is the best and the most accurate? For comparison, we have assembled in Table II the results of three investigations made by Barker and Winson,[1] and von Bergen, and comparing them with the theoretical value from the equation log $\frac{W}{L}$, where $\frac{W}{L}$ signifies the weight in milligrams of 10 meters of fibers at 18¼ per cent regain, we find quite an astonishing conformity of results. From the theoretical mathematical standpoint, the gravimetric method is the most accurate, but the practical application is limited to raw wool and top and can only be used for wools with no medulla. A further disadvantage is that it gives only the mean of the fineness and tells nothing about the variation within the sample. The cross-section method gives the geometric mean of the true diameter together with the correct frequency of size distribution.

Table II.—Results of Three Investigations Compared with the Theoretical Values.

Quality Appellation	S. G. Barker-Winson British Selected Tops			von Bergen U. S. Standard Top Sets Width Method[a]
	Cross-sectional Area Measurements	Gravimetric Method	Theoretical Value from the Equation log 10 $\frac{W}{L}$	
80's	19.2	19.6	19.2	19.5
70's	20.7	20.4	20.7	20.8
64's	21.4	22.3	22.4	21.9
60's	24.4	24.3	24.2	23.5
58's	26.0	25.7	26.2	24.8
56's	27.7	28.1	28.3	26.9
50's	31.5	31.1	30.6	30.4
48's	33.8	32.9	33.1	33.0
46's	35.1	....	....	34.8
44's	37.6	....	....	36.6
40's	38.2	....	....	38.3
36's	39.0	....	....	39.3
Number of fibers tested for quality	500	1000	....	500

[a] Samples taken from two Commercial Sets of U. S. Department of Agriculture.

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[1] S. G. Barker, "Wool Quality," pp. 134–138, His Majesty's Stationery Office, London (1931).

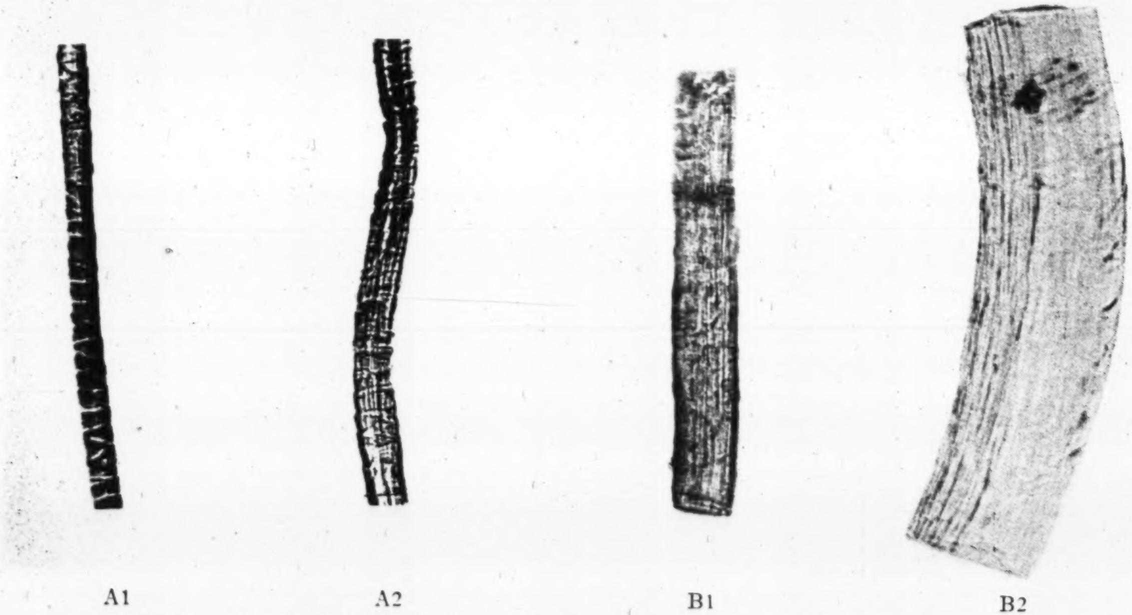

FIG. 16.—Caustic Soda Reaction on Sunlight-Damaged Wool (× 160). (Two specimens.)
(*A1* and *B1*) Before treatment.
(*A2* and *B2*) After treatment with NaOH.

From the mathematical standpoint the cross-section method is more accurate than the width method. As the diameter of the wool fiber varies not only from fiber to fiber, but the individual fiber itself is uneven over its length (see Fig. 6), the mean diameter found is only correct for the particular place where the fiber was measured.

In my opinion, for practical mill work, both the cross-section and width methods should be adopted as each has special advantages: (1) The cross-section method is necessary for the examination of the contour or degree of ellipticity and it is best suited for making standard photographs. (2) The width method is the simplest and the quickest to perform, and it is necessary for the longitudinal examination for the evenness of the fibers over the length and the condition of the epidermis.

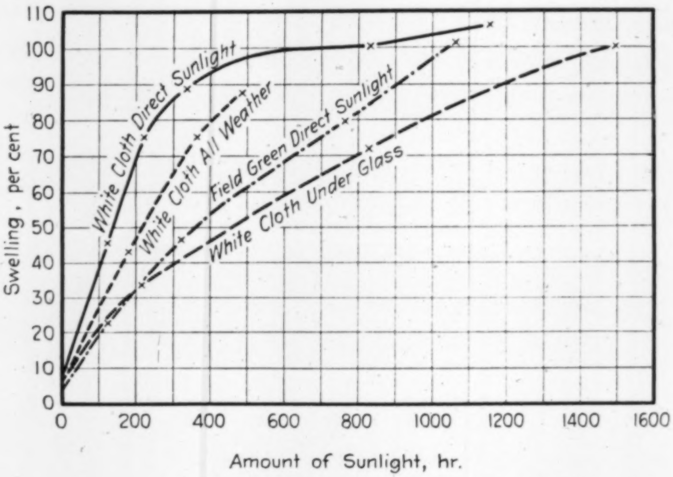

FIG. 17.—Curves Showing the Relation Between the Amount of Sunlight, Exposure Time and Swelling with 0.1 *N* NaOH on Wool, Taken out of Exposed Woolen Cloth.

DETERMINING THE EFFECT OF SUNLIGHT ON WOOL

Wool is affected through sunlight in its chemical and physical properties even while it is still on the animal. In the first weeks this is a bleaching effect, changing to a burning effect, due to the formation of acid from the sulfur present in the wool. The yellowish-brown discoloration and harsh feel of the upper ends of the staples from the back and the flanks of the animal is the result. The exposed fibers are brittle and show a high loss of strength. The affected fibers are much more sensitive than unaffected ones to chemicals, such as soda and caustic soda. Caustic soda has a strong swelling effect and under its influence the hairs start to curl. The effect of the sunlight also causes an increased or decreased affinity for certain dyestuffs.

The author[1] found that the extent of the swelling is, to a certain limit, in direct proportion to the length of exposure to sunlight. The difference in the width of the wool fiber before and after the swelling with caustic soda, expressed in per cent, can be used as a measure for the degree of damage.

The measuring of the width is done in the same manner as described under the width method for measuring fineness. The technique applied is as follows: The fibers to be tested are mounted in water, on a slide, and then the width measured. As soon as the first measurements are taken, two drops of 0.1 *N* NaOH are put near the rim of the cover glass. By means of filter paper the caustic soda is sucked over the fibers from the opposite side of the cover glass. This is repeated once more to be sure that all the fibers are in contact with the caustic alkali. The swelling takes place immediately and is completed in a few minutes, after which the second measurements of the width are then taken by measuring the same fibers at approximately the same place as in the first reading. Figure 16 illustrates this reaction.

TABLE III.—EFFECTS OF SUNLIGHT ON RAW WOOL.

Origin	Place on the Animal	End of the Staple		
		Width, Unswollen, μ	Width, Swollen, μ	Swelling, per cent
Australian wool	back	23.08	44.46	92.63
	belly	27.27	29.88	9.6
Virginia wool	back	35.06	45.23	29.00
	belly	42.08	44.37	5.44
Württemberg wool	back	33.49	43.42	29.65
	flanks	33.00	35.8	8.5
	belly	27.7	29.3	5.8
Swiss mountain wool	back	28.57	50.69	77.4

The relation between the amount of sunlight and the swelling with 0.1 *N* NaOH is illustrated best through the curves in Fig. 17. Through this relation the damage done to raw wool while still on the back of the sheep can be proved very clearly. Some of the results found on raw wool are given in Table III. For more detailed results reference should be made to the original published reports.

ACTION OF VARIOUS AGENTS ON WOOL AND THE DETERMINATION OF THEIR EFFECTS

Action of Hydrogen Peroxide:

Today most of the wool is bleached with hydrogen peroxide. It gives the best permanent white for woolen goods. In too-strong solutions the wool is very easily damaged by overbleaching or overoxidation. The injury to the fiber, when pronounced, can be seen under the microscope through the presence of crevices in the cortical layer. The caustic-soda reaction as described for testing the sunlight-damaged wool can also be used for the determination of damage by bleaching. In one case we found the percentage of swelling to be as follows:

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[1] Werner von Bergen, "Method of Determining the Effect of Light upon Wool," *The Melliand,* Vol. II, Nos. 1, 2 (1930).

	SWELLING, PER CENT
Unbleached	6.72
Bleached with 1 per cent hydrogen peroxide	17
Bleached with 3 per cent hydrogen peroxide	53.95

The last sample was completely stiff and had a glassy feel. Figure 18 shows three overbleached fibers.

Action of Chlorine:

Chlorine is used in the form of chlorinated lime. In a diluted chlorine solution, the wool is strangely altered in its properties. It becomes harsh, has a high luster, and acquires a silk-like feel, and at the same time loses its felting properties and the dyeing affinity is increased. The process of chlorination is employed principally in the printing of woolen fabrics and in the manufacturing of unshrinkable yarn for socks and sweaters. At the same time it finds application in lustering of oriental rugs and, also, of dulling the color somewhat so as to give an "antique" appearance.

The simplest and quickest method to decide whether a wool is chlorinated is the caustic soda reaction. If the chlorinated wool is treated under the microscope with a few drops of 0.1 *N* NaOH, it immediately swells up, over 100 per cent, as may be seen in Fig. 19.

When wool fibers are treated with concentrated chlorine water, the so-called Allword'sche reaction takes place. Along the hair, gas-filled bubbles like pearls are formed and the scales are dissolved. This reaction does not occur with an alkali-damaged fiber (see Fig. 20).

Action of Alkalies:

Wool is quite sensitive to alkalies. A 5 per cent solution of NaOH at a boiling temperature dissolves wool, completely, in a few minutes. Soda ash, potash and ammonia are not as destructive on wool, but should not be used at a temperature over 130 F. or in concentrated solution.

After being treated with an alkaline solution, woolen goods must be thoroughly washed in order to remove the last trace of alkali, as otherwise, after drying and storing, alkali spots may form, resulting in a weakening of the fiber and a discoloration of the goods.

The following microscopical tests (chemical) for testing alkali damage are in use:

Allword'sche Reaction.—This is described under action of chlorine on wool. The failure of this action to appear indicates alkali damage to

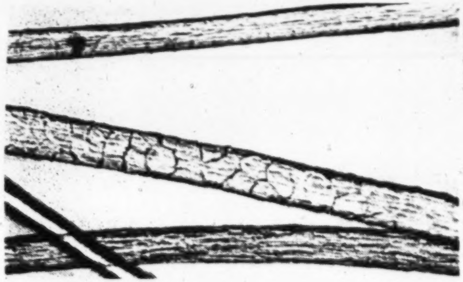

FIG. 18.—Wool Fibers Damaged Through Hydrogen Peroxide Bleaching (× 160).

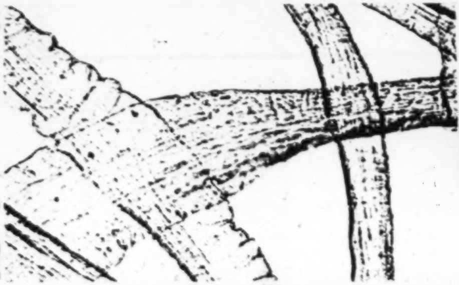

FIG. 19.—Chlorinated Wool Fibers After Treatment with 0.1 *N* NaOH (× 160).

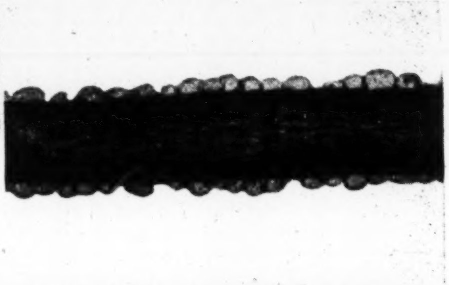

FIG. 20.—Wool Fibers After Treatment with Chlorine Water. Allword'sche Reaction (× 240).

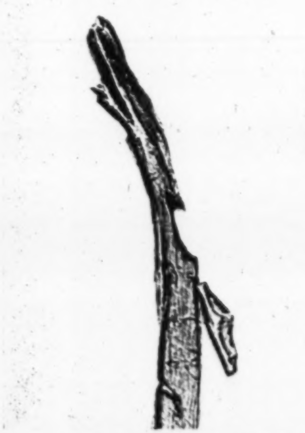

FIG. 21.—Wool Fiber Damaged Through Soda in Wool Scouring (× 160).

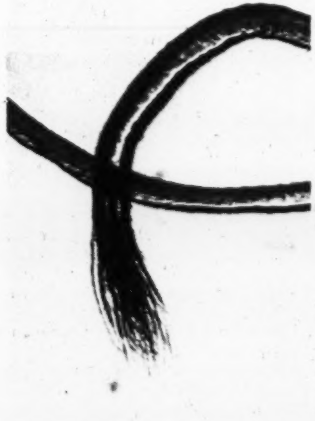

FIG. 22.—Wool Fiber Damaged Through Sulfuric Acid in Carbonizing (× 160).

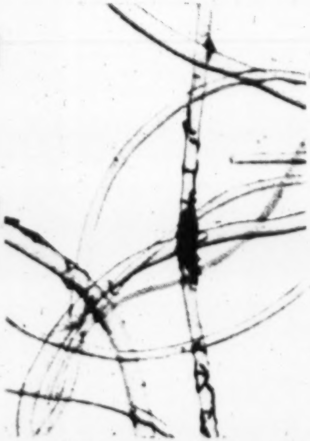

FIG. 23.—Fungoid Growths on Sized Wool Fibers (× 50).

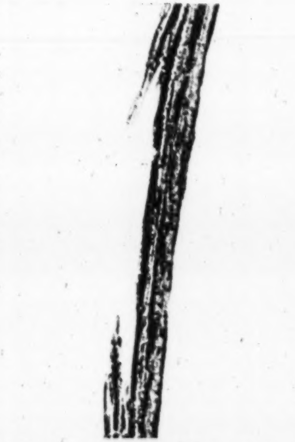

FIG. 24.—Wool Fiber Damaged Through Bacteria (× 160).

the wool of greater or lesser degree. Conclusions as to the quantitative degree of damage cannot be drawn since the reaction fails even when the reaction is only medium.

Diazo Reaction According to Pauly.—Injured wools are colored orange-yellow to a full red in a soda-alkaline diazo solution. The amino acid, tyrosin, present in the Veratin C of the fibrous layer yields this azo color reaction when coupled with diazo benzene sulfonic acid. Mark[1] then developed from this reaction a method which permits detection of injuries in the individual hairs qualitatively and to a certain extent also quantitatively.

This method was simplified by Sieber[2] who found that in place of the easily decomposed diazo benzene sulfonic acid the red direct dyestuff Benzopurpine 10B can be used. The diazo reaction is applicable only to white goods and its use is thus limited. Figure 21 shows a wool fiber damaged by alkali.

Action of Acids on Wool:

Up to the present time no satisfactory microscopical chemical test has been perfected for acid damage. The strongest damage in practical application of acid on wool is apt to occur in the carbonizing process. Figure 22 is an example of a damaged wool fiber through sulfuric acid carbonization. The epidermis is destroyed and the cortical layer is split open and the typical brush end is seen.

Mildew and Bacteria Damage:

Fungoid growths or mildew in wool can usually be detected without the use of the microscope, but for the proper identification the microscopic examination is necessary. In the manufacturing of woolen goods, fungoid growth is likely to occur mainly in sized material, which was loaded with starch or glue and became wet. The fungoid growth, as seen in Figure 23, was found on a sized warp. Usually no damage is done through mildew alone.

If wool is left in a warm place in a moist and alkali condition, so that the fiber does not have free access to plenty of fresh air, certain bacteria start to grow and produce enzymes, which rapidly break down the scales of the fiber and hydrolize the intercellular substance holding together the single cells of the cortical layer until they split open and fall apart (see Fig. 24).

The approximate bacteria damage can be estimated by counting the attacked fibers in a definite number of fibers and expressing it as a percentage.

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[1] H. Mark, "Beiträge zur Kenntnis der Woole und ihrer Bearbeitung," Gebrüder Borntraeger, Berlin (1925).

[2] W. Sieber, "Eine Prüfungs methode für Wollfaserschädigungen," *Melliand Textile Monthly*, p. 326 (1928).

For the determination of the resistance of wool fibers to bacterial disintegration, Burgess[1] suggests the following technique:

(*a*) ***Trypsin Solution***

Trypsin (B.D.H.)	0.5 g.
Water (distilled)	100 cc.

(*b*) ***Buffer Solution*** (pH value (8.6)

0.10 M Potassium di-hydrogen phosphate	34 cc.
0.05 M Borax	66 cc.

The wool (0.1 g.) is relieved of natural or added grease, wetted out in water and immersed in a mixture of 5 cc. each of solutions (*a*) and (*b*) in a test tube. Two drops of toluol are added and the tube corked and incubated at 35 to 40 C. Microscopical examinations of the wool are made first at hourly intervals and later after longer periods.

This method will be very useful to find out the variation in resistance to bacterial action between wool fibers and related fibers like mohair, cashmere and camel hair, which from the author's practical experience are much more sensitive to bacterial disintegration than the wool fiber itself.

CONCLUSION

This short review of the present knowledge of the structure of wool and of the several main physical and chemical properties cannot cover the subject completely, but it demonstrates the high value of microscopical research in the woolen field. It also shows that extensive research has so simplified these methods that they may be made a part of the daily routine work of any progressive mill laboratory. The determination of the wool fineness by the width method is already applied to wool top in some of the largest woolen mills in the country.

The cross-section method is applied in the selection of breeding animals for uniformity of fiber in wool production by the Bureau of Animal Industry, U. S. Department of Agriculture.

The method for the determination of sunlight damage may be used by the Army and Navy authorities to study the wearing properties of uniform material.

The two methods described for the determination of alkali-damaged wool are not conclusive enough and further research should be done in this direction.

Another field for research is to find the proper test for the determination of the action of acid. Mildew and bacteria damage are recognized quite clearly by the use of the microscope. A very useful method for the determination of resistance of wool and related fibers to bacterial action is that of Burgess.

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[1] R. Burgess, "The Use of Trypsin for the Determination of the Resistance of Wool Fibres to Bacterial Disintegration," *Journal*, Textile Inst., Vol. 25, p. T 289 (1934).

The Subcommittee on Wool of the Society's Committee D-13 on Textile Materials has made a start in the establishment of specifications and methods of testing the main physical dimensions of the wool fiber on the basis of the work described herein. The main goal is the setting up of a U. S. Quality Standard of Wool and Top consisting of 100 per cent domestic wool, which will be of immense value to the wool industry and its trade.

REFERENCES

NOTE.—No reference to the following is made in the text, but excerpts are taken therefrom and articles used as a guide:

W. F. Edwards, "Enzyme Action in the Textile Industries," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 725 (1932).

Harold De Witt Smith, "Some Aspects of the Structure and Properties of the Rayons," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 749 (1932).

J. M. Matthews, "The Textile Fibers," John Wiley and Sons, Inc., New York City (1924).

A MODIFIED SCHERING BRIDGE

BY W. B. KOUWENHOVEN,[1] P. H. DIKE[2] AND J. B. MCCURLEY[3]

SYNOPSIS

This paper describes a modified form of Schering bridge, completely shielded and guarded, which was developed by the Leeds & Northrup Co. and studied and improved in a joint investigation carried on by that company and the Electrical Laboratories of The Johns Hopkins University. This bridge possesses several novel features including a new method for balancing or compensating for the residual capacitance of the variable capacitor arm. It also uses a low voltage low impedance scheme for adjusting the shields and guards to the proper potential. The method of operation is discussed, the mathematical treatment is outlined, and certain experimental results are also given in this paper.

In Schering's[4] original bridge, Fig. 1, in which he recommended that R_3 be set to some convenient value and that R_4 and C_3 be varied to obtain balance, the power factor, δ_x, of the specimen C_x, is given by the expression:

$$\delta_x = \omega C_3 R_3$$

From this equation it is evident that the smallest power factor which can be measured with this arrangement is limited by the minimum or residual capacitance of C_3, provided ω and R_3 are fixed. One of the methods of overcoming this difficulty is to shunt the variable resistor R_4 with a capacitance[5] C_4. With this symmetrical arrangement the power factor equation becomes:

$$\delta_x = \omega C_3 R_3 - \omega C_4 R_4$$

If this symmetrical bridge is operated at a one to one ratio the equation simplifies to:

$$\delta_x = \omega (C_3 - C_4) R_3$$

This, however, is not usually the case and the more complicated expression is the one generally used in calculating the power factor.

In the modified Schering bridge shown schematically in Fig. 2, the neutralization of the residual capacitance of capacitor C_3 is obtained by the introduction of a capacitor C_a which is connected between the mid point of R_3 and the shields. This method of compensation makes it possible to operate the bridge as suggested by Schering and at the same time obtain the advantages of the symmetrical bridge.

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[2] Chief, Physical Division, Research Dept., Leeds & Northrup Co., Philadelphia, Pa.

[3] Graduate Student, Johns Hopkins University, Baltimore, Md.

[4] H. Schering, "Tätigkeitsbericht der Physikalische Technische Reichsanstalt," *Zeitschrift für Instrumentkunde*, Vol. 40, p. 124 (1920).

[5] E. Giebe and G. Zickner, "Verlustmessungen an Kondensatorem," *Archiv für Elektrotechnik*, Vol. 11, pp. 109–129 (1922).

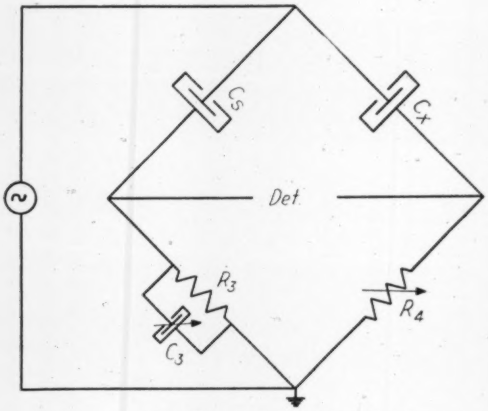

FIG. 1.—The Original Schering Bridge.

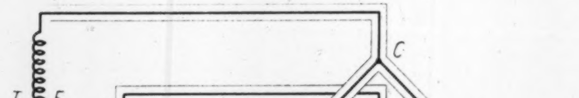

FIG. 2.—The Modified Schering Bridge.

In order to understand the operation of the bridge, consider Fig. 2 which shows two bridge circuits, one the Schering bridge and the other the auxiliary bridge. The completed bridge circuit shown in this figure is the modified Schering bridge and consists of the standard air capacitor C_s, the specimen C_x, and the two lower bridge arms AD and BD. This bridge is supplied with power from a high-tension transformer T_1. In addition there is shown an auxiliary bridge consisting of two equal ratio arms R_1 and R_2 and the AD and BD arms. This auxiliary bridge is fed with power from the low-tension transformer T_2.

Either bridge may be set up by the use of the switches, Sw_2, indicated in Fig. 2. For example, when the auxiliary bridge connection is desired, the operation of the switches Sw_2 disconnects C_s and C_x from the A and B corners of the bridge and connects the resistance arms R_1 and R_2 in their respective places. When the switches are thrown to the opposite position, R_1 and R_2 are cut out of the circuit and the Schering bridge is established.

The bridge is completely shielded and guarded as indicated in Fig. 2. These shields and the guard rings of the standard air capacitor and of the specimen are usually all connected to ground. At balance the A and B corners of the bridge are usually at ground potential. This is accomplished by bringing the potential of the D corner of the bridge to slightly below ground by means of the shield balancing circuit, which consists of a coarse resistor R, a slide wire resistor, r, and a mutual inductor m, all fed by a low-potential transformer T_3. This arrangement not only avoids the necessity for a high-voltage circuit for establishing the shield potentials but assures that all parts of the bridge circuit with which the operator may come in contact are either solidly grounded or grounded through a low impedance circuit. It also assures that the guard rings of the standard and test capacitors are at the potential of the low-voltage or measuring electrodes by simply connecting them to the shielding system of the bridge.

A switch Sw_1 is used to connect one side of the detector to either the shields or to the A corner of the bridge. The other terminal of the detector is permanently connected to the B corner of the bridge.

The same power source supplies the primaries of all three transformers T_1, T_2, and T_3. The connections are arranged, however, so that only two of the transformers are excited at any one time. When the auxiliary bridge is used, transformers T_2 and T_3 are excited; and with the Schering bridge in circuit T_1 and T_3 are energized.

Operation:

The complete operation of the modified Schering bridge is as follows:

(*a*) The auxiliary bridge is established by throwing switches Sw_2 to the proper positions. The capacitor C_3 is set at its zero scale reading or some convenient value, C_0. Transformers T_2 and T_3 are energized and with the detector connected to the point A, the bridge is balanced by adjusting R_4 and the capacitor C_a, which compensates for the lead and the initial or residual capacitance in the C_3 arm.

(*b*) The detector is then connected to the shields by operating switch Sw_1 and the potential of the D corner of the bridge is varied by adjustment of the resistor R, the sliding contact of resistor r, and the setting of the mutual inductor m until the detector shows that the shield and the B corner of the bridge are at the same potential.

(*c*) Repeat (*a*) and then (*b*) until both the auxiliary bridge and the shield balances hold simultaneously.

(*d*) Switches Sw_2 are then operated so as to substitute the capacitors C_s and C_x for the resistors R_1 and R_2 respectively. Transformer T_2 is disconnected and transformers T_1 and T_3 are energized. With the detector connected to the A corner of the bridge, balance is obtained by varying R_4 and C_3, let C_3 be the new value of the capacitance.

(*e*) The detector is then connected to the shields and R, r, and m are adjusted for balance.

(*f*) Repeat alternately (*d*) and (*e*) until simultaneous balances hold. This completes the balancing operation.

Under these conditions, assuming all elements pure, the power factor of the specimen is given by the expression:

$$\delta_x = \omega \Delta C_3 R_3 \qquad (1)$$

Here ΔC_3 represents the change in the capacitance of capacitor C_3 from its setting C_0 in the auxiliary bridge to the setting C_3 required to balance the bridge with the specimen in the circuit.

The capacitance, C_x, of the specimen is given by the relation:

$$C_x = C_s \frac{R_3}{R_4'}, \qquad (2)$$

Tests:

The bridge circuits were set up and tests were made to determine the characteristics of the bridge and the correctness of the theoretical calculations. In this work the detector used was the pointer galvanometer with amplifier. The sensitivity of the bridge with the detector available and at the voltages used was 0.00003 in power factor. The voltage supplied to the modified Schering bridge by transformer T_1 was 700 v. The voltage for the auxiliary bridge amounted to 5 v. The transformer T_3 gave 2.5 v. The source of power used in the work consisted of a 5 kva., 3 phase, 60 cycle pure sine wave generator.

EXPERIMENTAL RESULTS

1. *Guard—Bridge Corner Capacitance:*

An analysis of the bridge showed that the balance relation, Eq. 1, was free from the effect of capacitance between the shields and the A and B bridge corners. To verify this the following tests were made:

(*a*) The auxiliary bridge was first carefully balanced. A variable air capacitor having a range from 200 to 4200 $\mu\mu$f. was then placed in turn between the shields and the *A* and *B* corners of the bridge. This inserted capacitor was varied over its entire range without disturbing the balance.

(*b*) The same tests were repeated in the Schering bridge and gave the same result.

2. *Balance Equations All Elements Pure:*

(*a*) The two important relations for the auxiliary bridge are:

$$R_1R_4 = R_2R_3 \qquad (3)$$

and

$$C_0 = \frac{C_a}{4} \qquad (4)$$

The resistors used for R_1, R_2, and R_3 were nominally 10,000 ohms. In numerous balances over a period of more than a month the value of R_4 necessary for balance ranged between 9999 and 10,000.6 ohms. It is apparent, therefore, that the relation holds at least to one part in ten thousand. The variation in R_4 that was obtained may be attributed to such factors as changes in temperature, contact resistances, sensitivity, and to the personal element.

To check the relation $C_a = 4\ C_0$, both air and mica capacitors were used with the following results:

For E_2 = 5 v., C_3 and C_a precision air capacitors:

C_3	C_a	$C_a/4$
375 $\mu\mu$f.	1484 $\mu\mu$f.	371 $\mu\mu$f.
342 $\mu\mu$f.	1352 $\mu\mu$f.	338 $\mu\mu$f.
295 $\mu\mu$f.	1190 $\mu\mu$f.	297 $\mu\mu$f.

For E_2 = 2 volts, C_3 and C_a mica capacitors:

C_3	C_a	$C_a/4$
0.05 $\mu\mu$f.	0.20 $\mu\mu$f.	0.050 $\mu\mu$f.
0.05 $\mu\mu$f.	0.20 $\mu\mu$f.	0.050 $\mu\mu$f.

In these tests the capacitance of the leads was neglected. The results check Eq. 4.

(*b*) The correctness of bridge Eq. 2 was checked with the bridge operated at 400 v. and with precision type air capacitors forming both the C_s and C_x arms. During the test, C_s was held constant at 586 $\mu\mu$f. and C_x was varied. The results are given below:

R_3	R_4'	C_x	$\frac{C_xR_4'}{R_3}$	C_s
10 000 ohms	8 310 ohms	708 $\mu\mu$f.	588 $\mu\mu$f.	586 $\mu\mu$f.
10 000 ohms	10 020 ohms	587 $\mu\mu$f.	588 $\mu\mu$f.	586 $\mu\mu$f.
10 000 ohms	12 630 ohms	465 $\mu\mu$f.	587 $\mu\mu$f.	586 $\mu\mu$f.
10 000 ohms	17 090 ohms	343 $\mu\mu$f.	586 $\mu\mu$f.	586 $\mu\mu$f.

A comparison of the results in the last two columns shows clearly the correctness of the equation.

(*c*) The correctness of the power factor Eq. 1 was checked against the Low Power Factor Standard[1] of Kouwenhoven and Berberich. The standard capacitor consisted of a highly polished and cleaned flat brass plate air capacitor provided with guards. The measurements were made at 700 v. with the results given below. In every case they check to within the sensitivity of the bridge as used:

STANDARD OF POWER FACTOR	MEASURED POWER FACTOR	APPARENT ERROR
0.000000	0.0000*00*	0.0000*00*
0.000195	0.0002*15*	0.0000*20*
0.000996	0.0010*25*	0.0000*29*
0.001993	0.0019*99*	0.0000*06*

(*d*) The power factor Eq. 1 for the bridge is independent of the value of the variable resistance arm, R_4. To verify this a series of measurements of the Power Factor Standard was made at different bridge ratios. The results are given below for two sets of measurements. The results show that the measured values of power factor are independent of the ratio of C_s to C_x at least to within the sensitivity of the bridge and detector.

				POWER FACTOR STANDARD	
R_3, OHMS	R_4, OHMS	C_0, $\mu\mu$f.	C_3, $\mu\mu$f.	MEASURED δ_x	RATED VALUE δ_x
10 000	5 005.8	223	223	0.00000	0.000000
10 000	9 983.0	223	223	0.00000	0.000000
10 000	10 954.0	223	223	0.00000	0.000000
10 000	5 005.8	223	747	0.00197*8*	0.001993
10 000	9 983.0	223	750	0.0019*87*	0.001993
10 000	10 954.0	223	749	0.0019*83*	0.001993

(*e*) An investigation was made to determine the effect of the use of a capacitor in the C_a arm having an appreciable phase defect angle. In order to simulate this condition the variable air capacitor, C_a, which meggered infinity, was shunted by grid leaks of various values. When a five megohm grid leak was connected across C_a it produced a change of six parts in 10,000 in R_4. It is evident, therefore, that C_a should be a good air capacitor with high insulation resistance.

(*f*) A phase defect in R_3, since it will be of the nature of a capacitance, is compensated for by an increase in C_a. This is substantiated by the results given below for two separate measurements of the same specimen using different values of C_0:

$C_0 = 50\ \mu\mu\text{f.}$ $C_3 = 581\ \mu\mu\text{f.}$ P.F. = 0.0019*92*
$C_0 = 223\ \mu\mu\text{f.}$ $C_3 = 750\ \mu\mu\text{f.}$ P.F. = 0.0019*87*

One will note that the power factor obtained in both cases was the same to within the sensitivity of the bridge.

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[1] W. B. Kouwenhoven and L. J. Berberich, "A Standard of Low Power Factor," *Transactions*, Am. Inst. Electrical Engrs., Vol. 52, June, 1933, p. 521.

CONCLUSIONS

1. The modified Schering bridge presented in this paper had a sensitivity of 0.00003 for power factor when operated at 700 v. and 60 cycles with the available detector. This sensitivity should not by any means be considered the limiting one for this bridge since it may be improved by the use of either a higher voltage or a more sensitive detector.

2. The over-all sensitivity is limited by the less sensitive of the two bridges, either the auxiliary or the Schering. For this reason the voltage applied to the auxiliary bridge should be sufficiently high to give at least the same sensitivity to changes in the C_3 arm that exists in the Schering bridge.

3. The balance equations are shown to be correct at least to within the sensitivity employed.

4. The low impedance low-voltage shield balancing arrangement adds considerably to the safety of operation of this bridge compared to other Schering bridges employing the Wagner ground with its adjustable high-voltage element.

5. The guard rings of the standard and specimen need not be balanced separately but are brought to the proper potential by simply connecting them to the shields.

6. The method of compensating for the minimum or residual capacitance not only affords compensation at all ratios but also simplifies the calculations considerably.

7. No adjustable high voltage standard is required in this bridge.

8. The operation of the bridge is relatively simple and the shield balancing circuit is quite stable. Once properly made the auxiliary balance needs to be checked only occasionally.

[For Discussion, see page 744.—ED.]

THE TRANSFORMER BRIDGE

By J. B. McCurley,[1] W. B. Kouwenhoven[2] and P. H. Dike[3]

The Schering bridge has long been recognized for its accuracy and dependability in the measurement of the dielectric characteristics of insulating materials. Its successful operation requires considerable care and skill in manipulation, and for accurate work a carefully designed shield and guard system is necessary. This demands a double balance of the bridge by successive approximations, which makes the operation slow in routine testing.

To meet these objections a bridge for the measurement of dielectric characteristics at commercial frequencies has been devised and is described in this paper. This bridge is a Leeds & Northrup modification of the Doyle and Salter[4] transformer bridge, and has for its distinguishing features, rapidity and ease of operation, and a simple shielding arrangement which eliminates the complex guard balancing circuits.

The transformer bridge is not intended for use where the ultimate in accuracy and precision is required. It provides, however, an exceedingly simple and conveniently operated method which is at the same time reasonably accurate. The experimental bridge with 1000 volts applied to the specimen gave results which were accurate and reproducible to ±0.00003 in power factor on specimens of known characteristics.

General Description

The transformer bridge is shown in Fig. 1. A high-voltage transformer T_1, one end of which is grounded, supplies power for the standard C_s and the specimen C_x. A low-voltage transformer T_{3-4}, having a center tapped secondary with the center tap connected to ground, feeds the auxiliary arm consisting of a resistor R_4 and the balancing arm composed of a resistor R_3 in shunt with a mica capacitor C_3. Resistors R_3 and R_4 and capacitor C_3 are variable. The standard capacitor C_s is preferably a variable air capacitor with a negligible phase angle, though a fixed capacitor having a known phase angle may be used.

The detector is connected between ground and the common junction of R_3, C_3, and R_4 marked A in Fig. 1. A double pole, double throw switch not shown in the figure is used to interchange the standard and specimen; that is, to change the circuit from that shown in "Position A" to the one shown in "Position B" of Fig. 1. A study of this figure will show that C_x in position A and C_s in position B act simply as a load on transformer T_1. Their currents do not enter the galvanometer circuit; hence, the only influence they can have on the balance relation is by changing the voltage E_2 due to the regulation of T_1. Interchanging C_x and C_s, as mentioned above, keeps the load constant on transformer T_1 since in either position T_1 supplies both C_s and C_x. Therefore E_2 remains constant.

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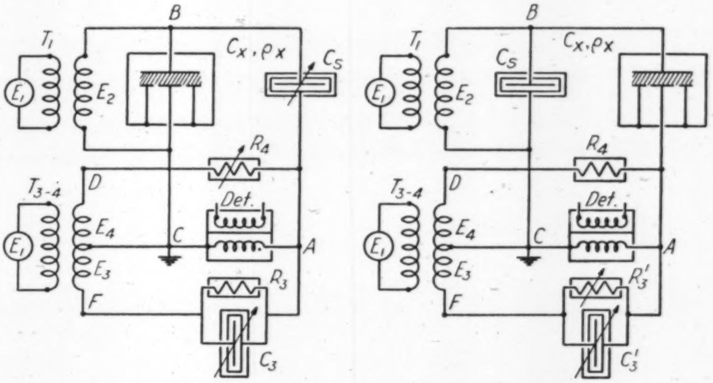
[2] Professor of Electrical Engineering, Johns Hopkins University, Baltimore, Md.

[3] Chief, Physical Division, Research Dept., Leeds & Northrup Co., Philadelphia, Pa.

[4] E. D. Doyle and E. H. Salter, "Methods of Measuring Dielectric Power Loss and Power Factor," *Transactions*, Am. Inst. Electrical Engrs., Vol. XLV, p. 630 (1926).

As shown and as used, a coupling transformer connects the bridge to the amplifier. If one side of the amplifier is connected to ground the coupling transformer may be dispensed with. This is a highly commendable feature since such a transformer may introduce both electrostatic and electromagnetic troubles which are sometimes extremely difficult to cope with.



Position A Position B

Note: All unconnected shields are grounded

FIG. 1.—The Transformer Bridge.

The flexibility of this bridge may be considerably increased by the insertion of a variable voltage transformer such as the Variac[1] to feed the primary of T_{3-4}. The reason for this will be discussed later. Briefly, it is used to control the magnitude of the voltages E_3 and E_4. If the Variac or some other device for varying the primary potential of T_{3-4} is not used both T_1 and T_{3-4} are fed directly from the same source, that is, they are both energized by E_1 as shown in Fig. 1.

The phase angle that exists between the voltage E_2 and the voltage $E_3 + E_4$ does not affect the results provided good transformers are used.

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[1] "The Variac—A New Adjustable Transformer," *The General Radio Experimenter*, Vol. VIII, Nos. 1 and 2, p. 5 (1933).

The bridge may be looked upon as made up of four main arms and an auxiliary circuit. The four arms are: (1) the secondary of transformer T_1; (2) C_s or C_x; (3) the lower half of the secondary of transformer T_{3-4}; and (4) R_3 and C_3 in parallel. The auxiliary circuit is made up of the other half of the secondary of transformer T_{3-4} and the resistor R_4 which serves to balance the conductance of the R_3, C_3 arm when C_s is in the circuit and part of this conductance when C_x is in the circuit.

All elements are shielded and all shields are tied to ground. If the standard and specimen have guards they may also be connected directly to ground, thus eliminating the necessity of elaborate guard balancing circuits.

OPERATION

There are a number of methods of operating the transformer bridge. These methods may be divided into two classifications: (1) operation with a variable standard C_s and (2) when equipment is limited operation with a fixed standard C_s and a fixed resistor R_4. In both classes there are two different methods of balancing. They are:

(*a*) Balance made with C_3, R_3, and R_4

(*b*) Balance made with C_3 and R_3 with R_4 fixed.

The operating procedure will now be given for these two methods.

Method 1 (a) C_s Variable, Balance Made with C_3, R_3, and R_4:

The method of operation is as follows:

With the elements arranged as shown in Fig. 1, position B, set R_3 at 10,000 ohms, or some other convenient value, and obtain a balance by adjusting C_3 and R_4.

Let their values be C_3' and R_4', respectively.

Switch to position A and rebalance with C_s and R_4 and let their values be C_s and R_4 respectively. During this operation R_3 and C_3 are not disturbed, that is, they still have values 10,000 ohms and C_3'.

Switch to position B and rebalance by changing R_3 to R_3'.

The constants of the unknown capacitor C_x are then given by the following equations:

$$C_x = C_s \qquad (1)$$

$$\delta_x = \frac{R_3 - R_3'}{\omega C_3' R_3' R_3} \qquad (2)$$

where δ_x = specimen power factor, and

$$\delta_x = \frac{10{,}000 - R_3'}{10{,}000\,\omega C_3' R_3'}, \text{ if } R_3 \text{ is set at 10,000 ohms as suggested above} \qquad (3)$$

These equations and all others used in this paper have been derived mathematically and their accuracy proven by experimental results. The mathematical analysis is entirely too long to be given in a paper of this nature where the average reader is not particularly concerned with the mathematical aspects but rather the field of application and utility of the bridge. A complete analysis may be found in the unabridged dissertation of McCurley.[1]

Method 2 (a), C_s Fixed, Balance Made with C_3, R_3, and R_4:

To operate, arrange elements as shown in Fig. 1, position A. Set $R_3 = 10{,}000$ ohms and balance the bridge by means of R_4 and C_3.

Switch to position B. Do not disturb R_4 and balance with C_3 and R_3. Let their new values be C_3' and R_3'.

Then

$$C_x = C_s \frac{C_3'}{C_3} \quad (4)$$

and

$$\delta_x = \frac{1}{\omega C_3'}\left(\frac{1}{R_3'} - \frac{K}{R_4}\right) - \frac{1}{\omega C_3}\left(\frac{1}{R_3} - \frac{K}{R_4}\right) \quad (5)$$

where $K = \frac{E_4}{E_3}$ the ratio of voltages across the points CD and CF respectively.

Method 1 (b), C_s Variable, R_4 Fixed, Balance Made with C_3 and R_3:

To operate, with elements arranged as shown in Fig. 1, position B, balance the bridge by means of R_3' and C_3'.

Shift to position A and rebalance by means of C_s and R_3, that is, change R_3' to R_3; leave C_3' undisturbed.

Under these conditions Eqs. 1 and 2 hold.

Method 2 (b), C_s and R_4 Fixed, Balance Made with C_3 and R_3:

To operate, arrange elements as shown in Fig. 1, position A, and balance bridge by means of R_3 and C_3.

Shift to position B and balance by changing R_3 to R_3' and C_3 to C_3'.

The balance equations for this method are Eqs. 4 and 5.

SHIELDING ARRANGEMENT

All the elements of the bridge are inclosed in grounded shields which eliminate entirely inter-element capacitances. The presence of these shields introduces capacitance to ground which act simply as loads on their respective transformers, as may be seen from a study of Fig. 1.

The specimen is provided with a guard electrode which is connected directly to ground. At balance the measuring electrode is also at ground potential and thus the necessity of an elaborate guard balancing circuit is entirely eliminated.

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[1] J. B. McCurley, "Methods of Measurement of Dielectric Characteristics at Commercial Frequencies," A Dissertation, Johns Hopkins University (1934).

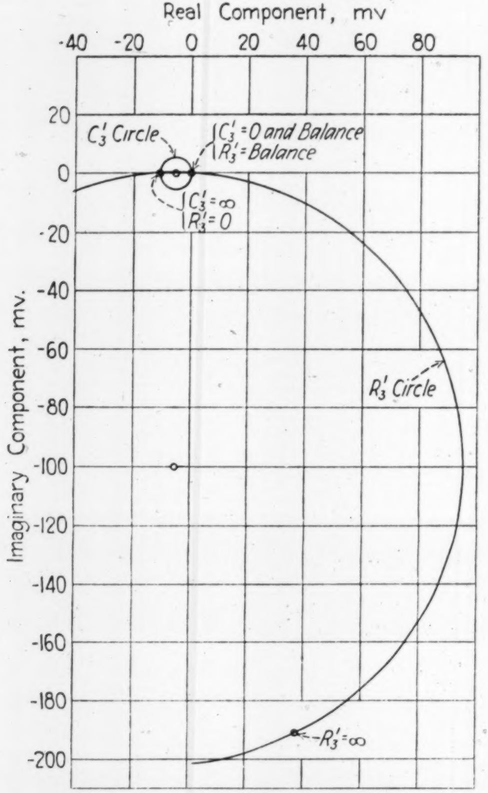

Fig. 2.—Variation of Galvanometer Potential $m = 100$.

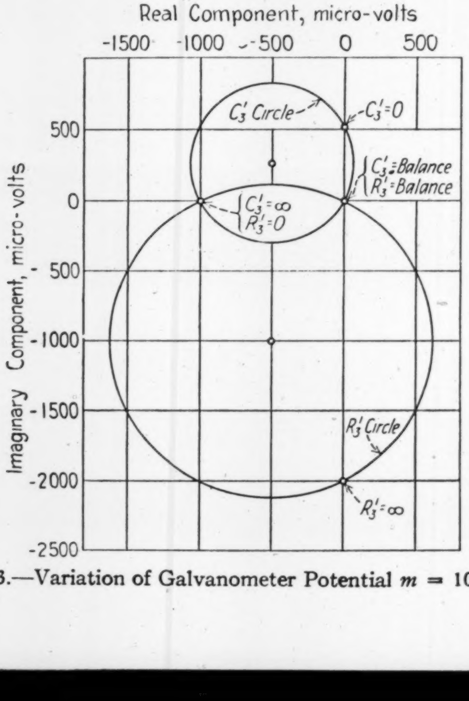

Fig. 3.—Variation of Galvanometer Potential $m = 1000$.

VARIATION OF POTENTIAL ACROSS GALVANOMETER

Figures 2 and 3 show the manner of variation of the potential across the detecting circuit as the balancing elements are varied over their complete range, starting from a condition of balance. Seletzky's[1] method of analysis was employed.

In both cases:

$$\delta_x = 0.01$$
$$\omega C_x = 50 \times 10^{-9} \qquad (C_x = 132. + \mu\mu\text{f.})$$
$$R_4 = 10^4$$

In Fig. 2 the ratio $E_2/E_3 = m$ was assumed as 100. The ratio m was 1000 for Fig. 3. Both figures are plotted in volts across the galvanometer circuit per volt of high-tension transformer.

One should note that in both cases the C_3' and R_3' circles are orthogonal, hence, an a.c. galvanometer in conjunction with a phase shifter, such as used by Kouwenhoven and Banos[2] is applicable for detecting unbalance in this bridge.

These circles show also that the bridge is quite sensitive to changes in resistance.

CONTROL OF MAGNITUDE $R_3 - R_3'$

Since $C_3' = mC_x$ where $m = E_2/E_3$, one may write

$$\delta_x = \frac{R_3 - R_3'}{\omega C_3' R_3' R_3} = \frac{R_3 - R_3'}{m\omega C_x R_3' R_3}$$

The numerator involves the difference, $R_3 - R_3'$, of two large numbers. For low power factors this difference may be quite small and, therefore, difficult to determine accurately.

One will note that for a given power factor the magnitude of $R_3 - R_3'$ varies directly with the capacitance C_x of the sample. One is usually restricted in the control of the magnitude of C_x and frequently C_x is definitely fixed. Hence, this method of increasing the difference is not particularly practicable. There are, however, two methods for controlling the magnitude of this difference:

1. Use 100,000 ohm instead of 10,000 ohm boxes in R_3 and R_4. This scheme will increase the difference a hundred fold. However, for the same quality of box the cost will increase. Kouwenhoven and Marks[3] have pointed out this same expedient in conjunction with the parallel resistance bridge.

2. The value of m may be increased by either lowering the value of

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[1] A. C. Seletzky, "Cross Potential of a 4-Arm Network," *Electrical Engineering*, December, 1933, p. 861.

[2] W. B. Kouwenhoven and A. Banos, Jr., "A High Sensitivity Power Factor Bridge," *Transactions*, Am. Inst. Electrical Engrs., Vol. 51, p. 202 (1932).

[3] W. B. Kouwenhoven and L. W. Marks, "A Comparison of the Methods for Testing Insulating Materials for Power Factor and Dielectric Constant at 1000 Cycles," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 821 (1934).

E_3 or increasing that of E_2. The former may be accomplished by means of a Variac or other potential controlling device, thus considerably increasing the flexibility of this bridge. Figures 2 and 3 show how a change in m influences the importance of R_3'. One will note that for $m = 1000$, $R_3 - R_3'$ is ten times as large as for $m = 100$.

EXPERIMENTAL

Effect of Shielding and Transformer Regulation:

It was stated under the section entitled shielding that any effective capacitance introduced by the shields is always directly across one of the three transformer secondaries, that is, across E_2, E_3, or E_4, and constitutes simply a load on transformers T_1 or $T_{3\text{-}4}$. To prove that such loads as would be introduced by any reasonable shielding arrangement have an entirely negligible effect, the following tests were made:

1. The bridge was carefully balanced with a sample in the circuit. A load of 1 μf., consisting of a mica capacitor, was then thrown across CF, Fig. 1, and did not upset the balance. This same load was then placed across CD with the same negative results.

2. A variable air capacitor was placed across BC and a load of 1300 μμf. was introduced without upsetting the balance in the least.

From these results one may safely conclude that any shielding arrangement within reason and likely to be encountered in practice will have no effect on the balance relations.

These results also show that for capacitance changes the regulation of the transformers used was not such as to cause an upset in the balance. As a further check on $T_{3\text{-}4}$ a variable resistor was placed across CF and it was found that this resistance could be lowered, without causing unbalance, to 4000 ohms for one transformer which we shall call A and 1500 ohms for another designated B. The resistor was then placed across CD and lowered to 3000 ohms for transformer A and 1000 ohms for transformer B without an upset in balance.

Now these burdens were in all cases far in excess of any contemplated in practice. Thus from these results one may conclude that for all power factors the regulations of the transformers may be neglected for samples of less than 1000 μμf. if the ratio of E_2 to E_3 is 1000. The reason for this statement lies in the fact that for this ratio a C_x of 1000 μμf. makes C_3 equal to 1 μf. Since it may seem to some that a sample of this size might cause an upset in T_1 let us recall the fact that a load of 1300 μμf. was thrown across E_2 without upsetting the balance.

Check of Balance Relations:

To check the validity of Eq. 2 the power factor standard of Kouwenhoven and Berberich[1] was used.

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[1] W. B. Kouwenhoven and L. J. Berberich, "A Standard of Low Power Factor," *Transactions*, Am. Inst. Electrical Engrs., Vol. 52, June, 1933, p. 521.

The following results were obtained:

$C_3 = C_3' = 0.522 \times 10^{-6}$ farads $\quad R_3 = 10{,}000$ ohms
$R_3' = 9961.3$ ohms and $\quad R_4 = 10{,}060$ ohms

From these values δ_x was calculated and found to be 0.001974 which compares favorably with the power factor of the standard, namely, 0.00199. The apparent error was less than 1 per cent.

Since R_3' could only be adjusted to within about 0.5 or 0.6 ohm with accuracy, this corresponds to a sensitivity of about ±0.00003 in power factor. Hence, the above apparent error may be attributed to this cause.

As a further check the Variac was changed three days later and the following results obtained:

$C_3 = C_3' = 0.5747 \times 10^{-6}$ farads $\quad R_3 = 10{,}000$ ohms
$R_3' = 9957.7$ ohms

This gives a power factor $\delta_x = 0.001965$ which again checks within the limit of sensitivity. The apparent error was 1.4 per cent.

For the next smaller value of the power factor standard, that is, 0.000996 the balance values were $C_3 = C_3' = 0.5747$ μf., $R_3 = 10{,}000$ ohms and $R_3' = 9979.6$ ohms from which the power factor $\delta_x = 0.000945$ or an apparent error of 5.1 per cent. It is not remarkable that this error is considerably larger than those obtained for the larger values of the power factor standard since the numerator of the expression for δ_x involves the term $R_3 - R_3'$ which in this case is only 20.4 ohms and a cumulative error in obtaining R_3 and R_3' amounting to 1 ohm will cause an error of 5 per cent for this low power factor.

As a check on the validity of the relation $\dfrac{C_x}{C_s} = \dfrac{C_3'}{C_3}$ given in Eq. 4, the standard was set at some convenient value and C_3 read; C_3 was then changed to C_3', and a new balance obtained by changing C_s to a new value C_x with the following results:

C_s	C_3	C_3'	C_x CALCULATED	C_x MEASURED
150 μμf.	0.1616 μf.	0.1305 μf.	121 μμf.	123 μμf.
154 μμf.	0.1636 μf.	0.1643 μf.	155 μμf.	154 μμf.
191 μμf.	0.2046 μf.	0.1744 μf.	163 μμf.	163 μμf.
240 μμf.	0.2590 μf.	0.2749 μf.	255 μμf.	254 μμf.
300 μμf.	0.3257 μf.	0.3128 μf.	288 μμf.	288 μμf.

In the above, C_x calculated was determined from Eq. 4 and C_x measured was the value obtained from the calibration chart of the standard C_s. Each scale division on C_s was equal to 6 μμf. and if we assume that the scale could be read accurately to a quarter of a division this would represent 1.5 μμf. All the readings given above fall within this tolerance with the exception of the first, but since this is only 0.5 μμf. greater than the tolerance expected one may safely conclude that the relation $C_x = \dfrac{C_s C_3'}{C_3}$ is correct.

As a check on Eq. 5 and to show that the various methods of operation give consistent results three samples having a wide range in power factor were measured with the following results:

Sample	Capacitance, μμf.	Method 1 (a)	Method 1 (b)	Method 2 (a)	Method 2 (b)	Mean Value	Maximum Deviation from Mean Value, per cent
No. 1.......	155	0.0517	0.0516	0.0517	0.0515	0.0516	0.194
No. 2.......	163	0.1760	0.1755	0.1755	0.1750	0.1755	0.285
No. 3.......	254	0.3220	0.3215	0.3212	0.3215	0.3216	0.124

The dielectric in sample No. 1 was paper laminated, in sample No. 2 was clear yellow bakelite, and in sample No. 3 was black bakelite, all commercial insulating materials.

In all cases samples were kept at constant temperature and humidity for at least 12 hours prior to taking readings. In Eq. 5, K was determined by means of a quadrant electrometer.

The power factor sensitivity used in the above was approximately 0.0005. This allowed an extremely quick and easily made balance such as the bridge is intended for.

Sensitivity and Accuracy

The degree to which the difference $R_3 - R_3'$ may be determined is the factor which controls both the sensitivity and accuracy of this bridge.

The sensitivity of the bridge as measured by calculating the power factor corresponding to the minimum positively detectable upset in R_3 was 0.00003. To obtain this sensitivity with the set-up used required careful adjustment of the bridge. The results obtained with the power factor standard show clearly that this sensitivity was realized.

These results show the sensitivity and accuracy that may be expected with careful operation. However, the bridge is intended to give a quick and easily operated method for the determination of the power factor of specimens such as sheet dielectrics. Operated in this manner the sensitivity will naturally be somewhat less.

Reproducibility of Results

The reproducibility of results and hence the reliability of the bridge may be shown by the fact that for about two months one sample showed the same value of measured power factor, 0.00284, to within the sensitivity used which was 0.00003. The sample was kept at a temperature of 25.1 C. and at a relative humidity of 62.5 per cent.

No change could be detected in the difference $R_3 - R_3'$ but during this time there was a change in C_3' of 0.01 per cent which is entirely negligible so far as its effect on power factor is concerned.

SUMMARY

The transformer bridge offers to a user a rapid and at the same time reasonably accurate method for the determination of dielectric characteristics. No guard balancing circuit is necessary and the shielding arrangement offers no difficulties. With fairly good equipment, phase angles in the elements, with the exception of the standard C_s, have a negligible influence on the balance relations. Two methods of controlling the magnitude of the difference $R_3 - R_3'$ have been pointed out.

The sensitivity and therefore the accuracy of the bridge was 0.00003 for power factor if operated carefully. Extremely rapid measurements could be made if an accuracy in power factor of only ±0.0005 was desired. There are a number of methods of operation available.

The preferred method of operation is method 1 (*a*) for in this all correction terms are eliminated and the calculations are the least involved. One pays for this advantage by the necessity of an additional balance and by the requirement of an adjustable standard capacitor. However, since a knowledge of *K* and the correct value of R_4, as is the case in Method 2 (*a*), are no longer required these sources of error vanish.

Methods 1 (*b*) and 2 (*b*) are almost as good as the above provided a balance can be obtained with the value of R_4 used. The outstanding advantages of the preferred method are: greater ease of calculation and possibility of balancing with a variable, R_4, whereas it might be impossible to balance with R_4 fixed.

DISCUSSION

Mr. H. L. Curtis.[1]—In discussing these papers there are one or two points that might well be brought out. The authors have introduced some very interesting methods for compensating certain features of the bridges, and these will take their places along with numerous other ones which are now being used in various places. The ones which are going to survive will be determined by factors which we cannot entirely foresee.

These bridges are useful in determining two different qualities of materials, namely, dielectric constant and power factor. In measuring the dielectric constant of a material, measurements of the mechanical dimensions are also required, but in measuring the power factor, no measurement of dimension is necessary.

For most of the materials for which power factor or dielectric constant is wanted, the uniformity of the material is not such that high accuracy is possible or required. There is no use in measuring with a higher accuracy than can be reproduced by the material. There are very few materials for which two samples will give the same result with much accuracy. Therefore, there is no necessity in most of our measurements for having bridges of great accuracy.

On the other hand, I think in the measurement of power factor particularly, there is a very great need for encouraging people to make measurements, as it is more or less of an exploratory field. We do not know whether or not improvements will result when measurements of the power factor of electric insulating materials become more general. Therefore, it seems to me, the methods should be kept as simple as possible and such that they can be used to a considerable extent.

I believe that the shielding which has come to be the fashionable thing in connection with these bridges adds to the complication and expense of the bridge, and that the increased accuracy is not warranted by the character of many of the materials that are measured. Mr. Kouwenhoven has circulated, to Committee D-9 on Electrical Insulating Materials, results which bear out this statement, and within a very few weeks Mr. A. H. Scott, one of my assistants, has obtained measurements by several different types of bridges, shielded, partially shielded, and unshielded. The variations in the results on a particular sample are not significant.

I hope that these types of measurements will be put on a basis where they can be extensively made with the accuracy that is really essential. There are some applications where very careful, precise, and elaborate bridges are probably necessary.

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[1] Principal Physicist, National Bureau of Standards, Washington, D. C.

I also wish to say that we are a long way off from the state where you can take a piece of apparatus and give it to any laborer and have him get a result that will mean something. It is still the case that if you are going to have power factor measurements that are of any value, you need to have some intelligence back of those measurements because there are too many factors that are still unknown to be able to grind out results.

In making measurements at power frequencies, a vibration galvanometer is a convenient detecting instrument to be used in these bridges. The instrument is rugged and very convenient in use, provided the frequency of the alternating current supply is maintained constant as is the case in communities where electric clocks are employed.

MR. E. J. RUTAN.[1]—Referring to the paper on the modified Schering bridge, the introduction of the capacitor C_a and tying it into the shield complicates the balance. Have the authors considered the possibility of using an inductometer, in view of the fact that its absolute value does not enter into the measurement obtained? If they have, why did they discard it?

MR. P. H. DIKE.[2]—Referring to Mr. Curtis's remarks on putting the measurement of power factor into the hands of unskilled observers, I would say that a completely shielded bridge can be much more safely operated by a relatively inexperienced observer than can an unshielded bridge. With the trained intelligence available at the Bureau of Standards, and with its stability of arrangement of equipment, it is possible to operate an unshielded bridge successfully, but the average commercial laboratory is unable to take the precautions requisite to make measurements which are consistent with each other without shielding.

With an unshielded bridge, even when a substitution method of measurement is employed, the power factor measurements are dependent on the surroundings of the bridge, the exact position of the operator, and the magnitudes of potentials in neighboring power leads. Even if these conditions are kept stable, we cannot be sure of getting the same power factor value for a given specimen in different laboratories. The best means of securing consistent results from different bridges is not to specify similar bridges, but to require that the bridges shall be adequately shielded.

Mr. Curtis remarks on the desirability of exploratory work on the measurement of power factor, and the encouragement of such work by making the apparatus and methods as simple as possible, and demanding no high degree of accuracy, until the significance of such measurements can be established. The transformer bridge as described lends itself admirably to such exploratory work, since it is simple in operation, completely shielded, requires no shield-balancing operation, and has an accuracy which is ample for the purpose. When the bridge is once built, the shielding can be forgotten as far as the observer is concerned, and the manipulation is almost as simple as that of a direct current bridge.

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[1] Superintendent, Test Bureau, The New York Edison Co., Inc., New York City.
[2] Chief, Physical Division, Research Dept., Leeds & Northrup Co., Philadelphia, Pa.

Mr. Curtis is entirely justified in saying that we are still a long way from the state where power factor measurements can be put in the hands of unskilled observers. He might well have said that the accuracy of power factor measurements is proportional to the intelligence factor of the man who makes them. This is certainly true where a high degree of precision is required as in measurements on cable oils.

As concerns Mr. Rutan's suggestion as to the use of an inductometer instead of the capacitor C_a, this expedient was the first to be considered. It was discarded mainly because of the fact that there is far more liability to pick-up of stray fields in an inductometer than in a capacitor, which introduces errors in balance which it is difficult to correct for or eliminate.

MR. W. B. KOUWENHOVEN[1] (*author's closure by letter*).—Mr. Curtis's remarks on the subject of shielding are very interesting. As Mr. Dike has pointed out it requires a very high grade of skill and special arrangements of the circuit to measure power factor accurately in a non-shielded bridge. It seems to me that it is best to operate shielded bridges where possible as that eliminates certain sources of error which may cause incorrect results.

I have made some experiments using an inductometer in place of a capacitor in shield balancing work. The presence of stray magnetic fields have always caused difficulty, therefore, where possible, I believe the use of inductometers should be avoided. It is well known that it is practically impossible to completely shield apparatus from the effects of a magnetic field. A capacitor, on the other hand, may be completely shielded by the use of very simple equipment.

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[1] Professor of Electrical Engineering, Johns Hopkins University, Baltimore, Md.

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